

Attachment 1

Project Scoping Meeting Minutes

Hunters Point Naval Shipyard Scoping Meeting San Francisco, California

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| ATTENDEES: | Derek Robinson, Navy Pat Brooks, Navy Bill Franklin, Navy Danielle Janda, Navy Lily Lee, EPA RPM David Yogi, EPA Jackie Lane, EPA Tamsen Drew, OCII (Office of Community Investment and Infrastructure) Amy Brownell, SFDPH Scott Hay, Cabrera Services Janet Naito, DTSC Nina Bacey, DTSC Sheetal Singh, CDPH | Jeff Wong, CDPH Tina Low, Water Board Kellie Koenig, CH2M Robert Kirkbright, CH2M Adam Engel, CH2M <i>On Phone:</i> Matt Slack, RASO Zach Edward, RASO LCDR Soric, RASO Dr. Steve Doremus, RASO Jana Dawson, Tech Law Mark Luckhardt, Five Point Lindsay Land, EPA Carla Brazen |
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DATE: December 13, 2016

PROJECT: Navy CLEAN 9000, CTO-FZ12

Objectives

The objectives of the meeting were to introduce team members, discuss radiological data evaluation and community outreach activities, and gain feedback, input, and buy-in from stakeholders.

Introduction

A presentation and schedule were provided to all invitees prior to the meeting.

Derek Robinson from the BRAC PMO kicked off the meeting by thanking everyone for attending, and stated how important this project is for the Navy, BRAC, the City of San Francisco, Regulatory Agencies, and developers. He stated the urgency of this effort and the requirement to get it done right the first time. Lily Lee mentioned the EPA will be sending a letter outlining recommended actions. Derek also said that this venue is a good place for everyone to meet face to face. Pat Brooks and CH2M will be presenting the strategy and scope of the planned efforts which hopefully can draw to a close any unanswered questions

Introductions were made.

The Tiger Team points of contact were identified:

NAVY - Pat Brooks, Derek Robinson, Danielle Janda, Zachary Edwards, and Matthew Slack

DTSC - Janet Niato and Nina Bacey

EPA - John Chestnut and Lily Lee

CDPH - Sheetal Singh and Jeff Wong

Water Board – Tina Low

City of San Francisco - Tamsen Drew (OCII) and Amy Brownell (SFDPH)

Bob Kirkbright began the presentation with a brief discussion of the team CH2M has assembled, pointing out the challenge in finding experts for the team that have not been involved with Tetra Tech or its affiliates and subcontractors to avoid any possible perception of a conflict of interest. The assembled team consists of a consortium of experienced experts from CH2M and other recognized radiological companies to provide an independent third party analysis of the data. He mentioned CH2M has been in contact with Dr. Covello to consult on the outreach messaging efforts; Dr. Covello indicated he would be available after the New Year.

Pat Brooks explained this project is BRAC's number one priority, and they will be putting all their efforts toward facilitating its completion.

Scott Hay presented what has been accomplished so far, and explained the technical approach to the project.

Pat Brooks reiterated the challenges of balancing the aggressive schedule with the thorough analysis this project demands. He indicated a substantial amount of rework has taken place, including past efforts by RASO and Tetra Tech, using approaches that identified anomalous data. It was mentioned there were additional accusations after those efforts were completed. The CH2M team will perform an independent analysis that will include reviewing those efforts as well as performing additional analysis of the data.

Technical Approach

Scott Hay presented the phased approach and accomplishments to date.

Questions were raised about the database regarding how it was going to be examined. Scott Hay and Pat Brooks explained the first steps are to determine the completeness of the data set. Sample IDs can be used to break out survey units, and arrange them by parcel to form data subsets.

A discussion was had about why 2006 was determined as the starting point. The EPA expressed concerns that Tetra Tech was working at Hunters Point in the 1990s and their Health Physicists have identified anomalies in some of the pre 2006 data that they reviewed. Pat Brooks explained that 2006 was used as the cutoff because that was when the TCRA to remove sewer lines began, and everything before that was characterization and preliminary surveys, not used to determine final status. It was decided that any data that was used for decision making needs to be reviewed, and Derek Robinson and Pat Brooks agreed to look into the data that was used to determine work was complete. EPA, DTSC, and the project team agreed if the pre 2006 data was superseded by other work done after 2006, it does not need to be analyzed.

A discussion was held regarding scope. It was explained that during the initial phase, only soil data will be reviewed. In later phases of the project, buildings scans and gamma statics will be evaluated as well. Items such as lab EDDs and field notebooks will be requested as needed as potential issues are identified.

Lily Lee expressed they have been getting a lot of questions on parcels that have already been transferred. Scott Hay explained that we are including all locations where Tetra Tech has worked, and analyzing all of their data. Further concerns were expressed regarding the data that does not show any obvious anomalies. It is her opinion that since Tetra Tech has disclosed that data has been falsified, we cannot say that the data is reliable even though the statistical tests do not turn up any results. Scott Hay and Bob Kirkbright explained that our statistical tests will identify anomalies in the data, including running tests designed to identify instances where data may have been falsified. It was agreed that areas of highest potential risk should be the priority.

Sheetal Singh asked questions about what types of tests were going to be run, and how it is known whether they are effective. Scott Hay explained we will be using a test on the data sets where problems have already been identified, as well as the data set in its entirety. If these tests are able to identify the known problem areas, it will provide confidence in the analysis. Scott Hay went on to describe the statistical test and how the analysis was going to be approached in more detail, and explained that phase two will determine the amount and locations of confirmation sampling.

EPA raised questions of the amount of confirmation sampling, and what approach will be taken if data testing methods do not recommend sampling in places where allegations have pointed to. Scott Hay explained that confirmation sampling will be done to address specific issues, including allegations from former workers, if that is deemed appropriate. It was discussed that allegations of misconduct do not necessarily mean that there is a health risk. Danielle Janda stated that the Navy was fully committed to doing a resampling effort, with the extent to be determined. Scott stated the North Pier will probably be used as a test run analysis.

Pat Brooks mentioned this initial effort will examine static gamma readings, building scans, and soil data; only the soil data will be included in the initial analysis with the target completion in January 2017. Laboratory electronic data and gamma walk-over data has been requested from TetraTech. During the discussion it was noted that split sample results are not in NIRIS, so those would have to be obtained separately from the agency that conducted the analysis.

Community Outreach

Kellie Koenig provided a handout of the proposed format for the Draft Radiological Community Engagement Communications Plan, and presented the Community Involvement objectives, approach, tasks, and schedule. The following was discussed:

The group recommended adding the Water Board and Non-Regulatory City departments to the list of stakeholders. Key stakeholders will be included from all available lists including the 2014 CIP.

Tamsen Drew stated that the City recommends four languages, and with the known local population recommended public documents be provided in English, Spanish, written Chinese, Samoan, and Tagalog.

The communication efforts will include preparation of and frequently updating a FAQ sheet with answers generated through the Tiger Team.

EPA and City of San Francisco representatives expressed the community is very interested in being informed and involved throughout the duration of the project. The topic of fact sheets and the subjects of each one were discussed. It was suggested that a third fact sheet be added between sheet one and two in order to inform public about initial findings and explaining how the Navy is going to proceed. David Yogi of EPA expressed importance of keeping the community involved throughout the process, not just telling them what we did after it was complete.

Derek Robinson expressed his desire to present at the Mayor's Hunters Point Shipyard Citizen's Advisory Committee (CAC). Tamsen Drew stated that the CAC would be interested.

Additional Public Outreach discussion yielded the ideas that will be discussed further:

- Be proactive, not reactive.
- Get the community involved early in the process and bring them along the process to build trust.
- The possibility of EPA getting a third party technical advisor to help communicate technical aspect to the public. Agency grant availability information should be communicated to CBOs.
- Multiple feedback mechanisms for public communication are beneficial.

- Respond as quickly as possible to community concerns and give consistent responses; essential to building trust.
- Create a list of FAQs to facilitate fast and consistent responses to community questions.
- Go to reporters directly to get them involved, so they do not misinterpret what is going on. The Navy has specific reporters they have worked with in the past.
- Local “door to door” outreach has been successful in the past. Coordination with local churches and community groups has also been successful. A community liaison may help facilitate.
- Choose venues that facilitate the open exchange of information.
- Present Navy, EPA, State, City as a unit.
- Look into attending preexisting meetings
- Public stakeholders prefer information via Email.
- The District Supervisor expressed an interest to be involved.
- The community outreach team resolved to have a call twice per month for two months.

Tamsen Drew raised concern about CH2M’s past involvement at Hunters Point, and how the community may react to having a company with past history at the site doing the third party evaluation. Bob Kirkbright explained the differences in CH2M’s history and what occurred with Tetra Tech. It is recognized that a cohesive message is necessary to explain how the situations were vastly different, including the response by the companies; the CH2M team performing this review will include recognized senior experts from at least three other independent companies; and this effort will receive continuous independent scrutiny by the Federal, State, and Tiger Team members.

David Yogi of the EPA suggested that there are groups that will never change their distrust level and efforts are better spent on stakeholders who want to hear the facts and learn about current activities. He also brought up having a Technical Advisor, separate from CH2M and the Navy. Recommendations included Saul Bloom, and Kai Vetter. Pat Brooks commented that the Navy is working to involve a National Laboratory (such as Argonne National Laboratory), but it has not been contracted due to the time it takes to get them on board.

Bill Franklin discussed 3 key points:

- Need to identify the best forums and look for reasons to say “Yes” to outreach opportunities and venues to exchange information.
- Tiger Team to share public inquiries and answers to ROIs with the group to ensure a consistent message.
- Tiger Team participation in outreach and outreach planning meetings so that stakeholder interaction is productive and respectful.

Action Items

Determine if pre 2006 data was used for decision making – Pat Brooks

Provide library of compiled questions and answers on community outreach to share with team – Lily Lee

Plan twice a month Community outreach team check in meeting – Derek Robinson

Email copy of Draft Radiological Community Engagement Plan Communications Plan to RASO – Kellie Koenig

Attachment 2

Field SOPs

Application and Description of Standard Operating Procedures

| SOP Number | SOP Title | Application and Purpose |
|---------------|---|--|
| CH2M Document | Soil Sampling | Provides guidelines for obtaining samples of surface and subsurface soils using hand and drilling-rig-mounted equipment. |
| CH2M Document | Logging of Soil Borings | Provides guidance for obtaining accurate and consistent descriptions of soil characteristics during soil sampling operations. |
| CH2M Document | Locating and Clearing Underground Utilities | Provides general guidelines and specific procedures that must be followed when locating underground utilities and clearing dig locations. |
| CH2M Document | Decontamination of Equipment and Samples | Provides general guidelines for the decontamination of sampling equipment, and monitoring equipment used in potentially contaminated environments. |
| CH2M Document | Preparing Field Logbooks | Provides general guidelines for entering field data into logbooks during site investigation and remediation activities. |
| CH2M Document | Chain-of-Custody | Provides information on chain-of-custody procedures. |
| CH2M Document | Packaging and Shipping Procedures for Low-concentration Samples | Provides information on preparing, packaging, and shipping low activity radioactive samples for analysis. |

Soil Sampling

I. Purpose and Scope

The purpose of this procedure is to provide guidelines for obtaining samples of surface and subsurface soils using hand and drilling-rig mounted equipment.

II. Equipment and Materials

- Stainless-steel trowel, shovel, scoop, coring device, hand auger, or other appropriate hand tool
- Stainless-steel, split-spoon samplers
- Thin-walled sampling tubes
- Drilling rig or soil-coring rig
- Stainless-steel pan/bowl or disposable sealable bags
- Sample bottles

III. Procedures and Guidelines

Before sampling begins, equipment will be decontaminated using the procedures described in SOP *Decontamination of Drilling Rigs and Equipment*. The sampling point is located and recorded in the field logbook. Debris should be cleared from the sampling location.

A. Surface and Shallow Subsurface Sampling

A shovel, post-hole digger, or other tool can be used to remove soil to a point just above the interval to be sampled. A decontaminated sampling tool will be used to collect the sample when the desired sampling depth has been reached. Soil for semivolatile organic and inorganic analyses is placed in the bowl and mixed; soil for volatile organic analysis is not mixed or composited but is placed directly into the appropriate sample bottles. A stainless-steel or dedicated wooden tongue depressor is used to transfer the sample from the bowl to the container.

The soils removed from the borehole should be visually described in the field log book, including approximated depths.

When sampling is completed, photo-ionization device (PID) readings should be taken directly above the hole, and the hole is then backfilled.

More details are provided in the SOP *Shallow Soil Sampling*.

B. Split-Spoon Sampling

Using a drilling rig, a hole is advanced to the desired depth. For split-spoon sampling, the samples are then collected following the ASTM D 1586 standard (attached). The sampler is lowered into the hole and driven to a depth equal to the total length of the sampler; typically this is 24 inches. The sampler is driven in 6-inch increments using a 140-pound weight (“hammer”) dropped from a height of 30 inches. The number of hammer blows for each 6-inch interval is counted and recorded. To obtain enough volume of sample for subsequent laboratory analysis, use of a 3-inch ID sampler may be required. Blow counts obtained with a 3-inch ID spoon would not conform to ASTM D 1586 and would therefore not be used for geotechnical evaluations.

Once retrieved from the hole, the sampler is carefully split open. Care should be taken not to allow material in the sampler to fall out of the open end of the sampler. To collect the sample, the surface of the sample should be removed with a clean tool and disposed of. Samples collected for volatiles analysis should be placed directly into the sample containers from the desired depth in the split spoon. Material for samples for all other parameters should be removed to a decontaminated stainless steel tray or disposable sealable bag. The sample for semivolatile organic and inorganic analyses should be homogenized in the field by breaking the sample into small pieces and removing gravel. The homogenized sample should be placed in the sample containers. If sample volume requirements are not met by a single sample collection, additional sample volume may be obtained by collecting a sample from below the sample and compositing the sample for non-volatile parameters only.

Split-spoon samples also will be collected using a tripod rig. When using a tripod rig the soil samples are collected using an assembly similar to that used by the drilling rig.

C. Thin-Walled Tube Sampling

Undisturbed fine grained samples may be collected for analysis for geotechnical parameters such as vertical hydraulic conductivity. These samples will be collected using thin-walled sampling tubes (sometimes called Shelby tubes) according to ASTM D 1587 (attached). Tubes will be 24- to 36 inches long and 3- to 4-inches in diameter, depending upon the quantity of sample required. Undisturbed samples will be obtained by smoothly pressing the sampling tube through the interval to be sampled using the weight of the drilling rig. Jerking the sample should be avoided. Once the sample is brought to the surface, the ends will be sealed with bees wax and then sealed with end caps and heavy tape. The sample designation, data and time of sampling, and the up direction will be noted on the sampling tube. The tube shall be kept upright as much as possible and will be protected from freezing, which could disrupt the undisturbed nature of the sample. Samples for geochemical analysis normally are not collected from thin-walled tube samples.

IV. Attachments

ASTM D 1586 Standard Penetration Test Method for Penetration Test and Split-Barrel Sampling of Soils (ASTM D1586.pdf)

ASTM D 1587 Standard Practice for Thin-Walled Tube Sampling of Soils (ASTM D1587.pdf)

V. Key Checks and Preventative Maintenance

- Check that decontamination of equipment is thorough.
- Check that sample collection is swift to avoid loss of volatile organics during sampling.



Designation: D 1586 – 08

Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils¹

This standard is issued under the fixed designation D 1586; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method describes the procedure, generally known as the Standard Penetration Test (SPT), for driving a split-barrel sampler to obtain a representative disturbed soil sample for identification purposes, and measure the resistance of the soil to penetration of the sampler. Another method (Test Method D 3550) to drive a split-barrel sampler to obtain a representative soil sample is available but the hammer energy is not standardized.

1.2 Practice D 6026 gives a guide to determining the normalized penetration resistance of sands for energy adjustments of N-value to a constant energy level for evaluating liquefaction potential.

1.3 Test results and identification information are used to estimate subsurface conditions for foundation design.

1.4 Penetration resistance testing is typically performed at 5-foot depth intervals or when a significant change of materials is observed during drilling, unless otherwise specified.

1.5 This test method is limited to use in nonlithified soils and soils whose maximum particle size is approximately less than one-half of the sampler diameter.

1.6 This test method involves use of rotary drilling equipment (Guide D 5783, Practice D 6151). Other drilling and sampling procedures (Guide D 6286, Guide D 6169) are available and may be more appropriate. Considerations for hand driving or shallow sampling without boreholes are not addressed. Subsurface investigations should be recorded in accordance with Practice D 5434. Samples should be preserved and transported in accordance with Practice D 4220 using Group B. Soil samples should be identified by group name and symbol in accordance with Practice D 2488.

1.7 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D 6026, unless superseded by this test method.

1.8 The values stated in inch-pound units are to be regarded as standard, except as noted below. The values given in

parentheses are mathematical conversions to SI units, which are provided for information only and are not considered standard.

1.8.1 The gravitational system of inch-pound units is used when dealing with inch-pound units. In this system, the pound (lbf) represents a unit of force (weight), while the unit for mass is slugs.

1.9 Penetration resistance measurements often will involve safety planning, administration, and documentation. This test method does not purport to address all aspects of exploration and site safety. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Performance of the test usually involves use of a drill rig; therefore, safety requirements as outlined in applicable safety standards (for example, OSHA regulations,² NDA Drilling Safety Guide,³ drilling safety manuals, and other applicable state and local regulations) must be observed.

2. Referenced Documents

2.1 ASTM Standards:⁴

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

D 1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes

D 2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D 2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)

D 2488 Practice for Description and Identification of Soils

² Available from Occupational Safety and Health Administration (OSHA), 200 Constitution Ave., NW, Washington, DC 20210, <http://www.osha.gov>

³ Available from the National Drilling Association, 3511 Center Rd., Suite 8, Brunswick, OH 44212, <http://www.ndafo.com>.

⁴ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

¹ This method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Evaluations.

Current edition approved Feb. 1, 2008. Published March 2008. Originally approved in 1958. Last previous edition approved in 1999 as D 1586 – 99.

*A Summary of Changes section appears at the end of this standard.

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(Visual-Manual Procedure)

- D 3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils
- D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
- D 4220 Practices for Preserving and Transporting Soil Samples
- D 4633 Test Method for Energy Measurement for Dynamic Penetrometers
- D 5434 Guide for Field Logging of Subsurface Explorations of Soil and Rock
- D 5783 Guide for Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
- D 6026 Practice for Using Significant Digits in Geotechnical Data
- D 6066 Practice for Determining the Normalized Penetration Resistance of Sands for Evaluation of Liquefaction Potential
- D 6151 Practice for Using Hollow-Stem Augers for Geotechnical Exploration and Soil Sampling
- D 6169 Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations
- D 6286 Guide for Selection of Drilling Methods for Environmental Site Characterization
- D 6913 Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis

3. Terminology

3.1 *Definitions:* Definitions of terms included in Terminology D 683 specific to this practice are:

3.1.1 *cathead, n*—the rotating drum or windlass in the rope-cathead lift system around which the operator wraps a rope to lift and drop the hammer by successively tightening and loosening the rope turns around the drum.

3.1.2 *drill rods, n*—rods used to transmit downward force and torque to the drill bit while drilling a borehole.

3.1.3 *N-value, n*—the blow count representation of the penetration resistance of the soil. The *N-value*, reported in blows per foot, equals the sum of the number of blows (*N*) required to drive the sampler over the depth interval of 6 to 18 in. (150 to 450 mm) (see 7.3).

3.1.4 *Standard Penetration Test (SPT), n*—a test process in the bottom of the borehole where a split-barrel sampler having an inside diameter of either 1-1/2-in. (38.1 mm) or 1-3/8-in. (34.9 mm) (see Note 2) is driven a given distance of 1.0 ft (0.30 m) after a seating interval of 0.5 ft (0.15 m) using a hammer weighing approximately 140-lbf (623-N) falling 30 ± 1.0 in. (0.76 m \pm 0.030 m) for each hammer blow.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *anvil, n*—that portion of the drive-weight assembly which the hammer strikes and through which the hammer energy passes into the drill rods.

3.2.2 *drive weight assembly, n*—an assembly that consists of the hammer, anvil, hammer fall guide system, drill rod attachment system, and any hammer drop system hoisting attachments.

3.2.3 *hammer, n*—that portion of the drive-weight assembly consisting of the 140 ± 2 lbf (623 \pm 9 N) impact weight which is successively lifted and dropped to provide the energy that accomplishes the sampling and penetration.

3.2.4 *hammer drop system, n*—that portion of the drive-weight assembly by which the operator or automatic system accomplishes the lifting and dropping of the hammer to produce the blow.

3.2.5 *hammer fall guide, n*—that part of the drive-weight assembly used to guide the fall of the hammer.

3.2.6 *number of rope turns, n*—the total contact angle between the rope and the cathead at the beginning of the operator's rope slackening to drop the hammer, divided by 360° (see Fig. 1).

3.2.7 *sampling rods, n*—rods that connect the drive-weight assembly to the sampler. Drill rods are often used for this purpose.

4. Significance and Use

4.1 This test method provides a disturbed soil sample for moisture content determination, for identification and classification (Practices D 2487 and D 2488) purposes, and for laboratory tests appropriate for soil obtained from a sampler that will produce large shear strain disturbance in the sample such as Test Methods D 854, D 2216, and D 6913. Soil deposits containing gravels, cobbles, or boulders typically result in penetration refusal and damage to the equipment.

4.2 This test method provides a disturbed soil sample for moisture content determination and laboratory identification. Sample quality is generally not suitable for advanced laboratory testing for engineering properties. The process of driving the sampler will cause disturbance of the soil and change the engineering properties. Use of the thin wall tube sampler (Practice D 1587) may result in less disturbance in soft soils. Coring techniques may result in less disturbance than SPT sampling for harder soils, but it is not always the case, that is, some cemented soils may become loosened by water action during coring; see Practice D 6151, and Guide D 6169.

4.3 This test method is used extensively in a great variety of geotechnical exploration projects. Many local correlations and widely published correlations which relate blow count, or *N-value*, and the engineering behavior of earthworks and foundations are available. For evaluating the liquefaction potential of sands during an earthquake event, the *N-value* should be normalized to a standard overburden stress level. Practice D 6066 provides methods to obtain a record of normalized resistance of sands to the penetration of a standard sampler driven by a standard energy. The penetration resistance is adjusted to drill rod energy ratio of 60 % by using a hammer system with either an estimated energy delivery or directly measuring drill rod stress wave energy using Test Method D 4633.

Note 1—The reliability of data and interpretations generated by this practice is dependent on the competence of the personnel performing it

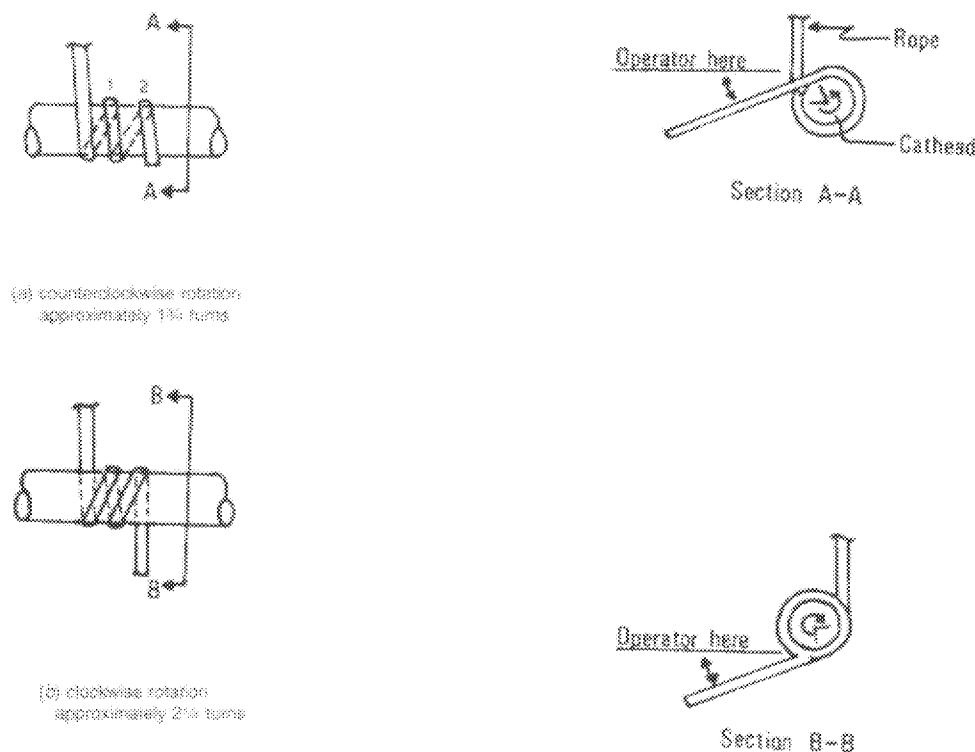


FIG. 1 Definitions of the Number of Rope Turns and the Angle for (a) Counterclockwise Rotation and (b) Clockwise Rotation of the Cathead

and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 generally are considered capable of competent testing. Users of this practice are cautioned that compliance with Practice D 3740 does not assure reliable testing. Reliable testing depends on several factors and Practice D 3740 provides a means of evaluating some of these factors. Practice D 3740 was developed for agencies engaged in the testing, inspection, or both, of soils and rock. As such, it is not totally applicable to agencies performing this practice. Users of this test method should recognize that the framework of Practice D 3740 is appropriate for evaluating the quality of an agency performing this test method. Currently, there is no known qualifying national authority that inspects agencies that perform this test method.

5. Apparatus

5.1 Drilling Equipment—Any drilling equipment that provides at the time of sampling a suitable borehole before insertion of the sampler and ensures that the penetration test is performed on undisturbed soil shall be acceptable. The following pieces of equipment have proven to be suitable for advancing a borehole in some subsurface conditions:

5.1.1 Drag, Chopping, and Fishtail Bits, less than 6½ in. (165 mm) and greater than 2¼ in. (57 mm) in diameter may be used in conjunction with open-hole rotary drilling or casing-advancement drilling methods. To avoid disturbance of the underlying soil, bottom discharge bits are not permitted; only side discharge bits are permitted.

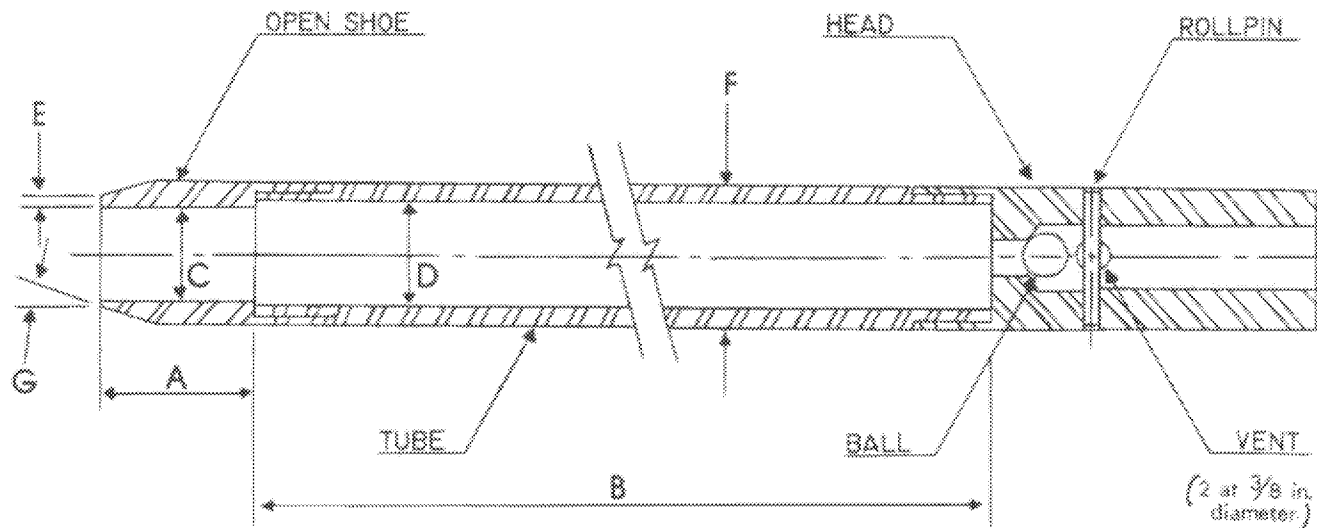
5.1.2 Roller-Cone Bits, less than 6½ in. (165 mm) and greater than 2¼ in. (57 mm) in diameter may be used in conjunction with open-hole rotary drilling or casing-advancement drilling methods if the drilling fluid discharge is deflected.

5.1.3 Hollow-Stem Continuous Flight Augers, with or without a center bit assembly, may be used to drill the borehole. The inside diameter of the hollow-stem augers shall be less than 6½ in. (165 mm) and not less than 2¼ in. (57 mm).

5.1.4 Solid, Continuous Flight, Bucket and Hand Augers, less than 6½ in. (165 mm) and not less than 2¼ in. (57 mm) in diameter may be used if the soil on the side of the borehole does not cave onto the sampler or sampling rods during sampling.

5.2 Sampling Rods—Flush-joint steel drill rods shall be used to connect the split-barrel sampler to the drive-weight assembly. The sampling rod shall have a stiffness (moment of inertia) equal to or greater than that of parallel wall “A” rod (a steel rod that has an outside diameter of 1-5/8 in. (41.3 mm) and an inside diameter of 1-1/8 in. (28.5 mm)).

5.3 Split-Barrel Sampler—The standard sampler dimensions are shown in Fig. 2. The sampler has an outside diameter of 2.00 in. (50.8 mm). The inside diameter of the of the split-barrel (dimension D in Fig. 2) can be either 1½-in. (38.1



A = 1.0 to 2.0 in. (25 to 50 mm)
 B = 18.0 to 30.0 in. (0.457 to 0.762 m)
 C = 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
 D = 1.60 ± 0.05 – 0.00 in. (38.1 ± 1.3 – 0.0 mm)
 E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
 F = 2.00 ± 0.05 – 0.00 in. (50.8 ± 1.3 – 0.0 mm)
 G = 16.0° to 22.0°

FIG. 2 Split-Barrel Sampler

mm) or 1½-in. (34.9 mm) (see Note 2). A 16-gauge liner can be used inside the 1½-in. (38.1 mm) split barrel sampler. The driving shoe shall be of hardened steel and shall be replaced or repaired when it becomes dented or distorted. The penetrating end of the drive shoe may be slightly rounded. The split-barrel sampler must be equipped with a ball check and vent. Metal or plastic baskets may be used to retain soil samples.

NOTE 2—Both theory and available test data suggest that *N*-values may differ as much as 10 to 30 % between a constant inside diameter sampler and upset wall sampler. If it is necessary to correct for the upset wall sampler refer to Practice D1086. In North America, it is now common practice to use an upset wall sampler with an inside diameter of 1½ in. At one time, liners were used but practice evolved to use the upset wall sampler without liners. Use of an upset wall sampler allows for use of retainers if needed, reduces inside friction, and improves recovery. Many other countries still use a constant ID split-barrel sampler, which was the original standard and still acceptable within this standard.

5.4 Drive-Weight Assembly

5.4.1 Hammer and Anvil—The hammer shall weigh 140 ± 2 lbf (623 ± 9 N) and shall be a rigid metallic mass. The hammer shall strike the anvil and make steel on steel contact when it is dropped. A hammer fall guide permitting an unimpeded fall shall be used. Fig. 3 shows a schematic of such hammers. Hammers used with the cathead and rope method shall have an unimpeded over lift capacity of at least 4 in. (100 mm). For safety reasons, the use of a hammer assembly with an internal anvil is encouraged as shown in Fig. 3. The total mass of the hammer assembly bearing on the drill rods should not be more than 250 ± 10 lbf (113 ± 5 kg).

NOTE 3—It is suggested that the hammer fall guide be permanently marked to enable the operator or inspector to judge the hammer drop height.

5.4.2 Hammer Drop System—Rope-cathead, trip, semi-automatic or automatic hammer drop systems, as shown in Fig. 4 may be used, providing the lifting apparatus will not cause penetration of the sampler while re-engaging and lifting the hammer.

5.5 Accessory Equipment—Accessories such as labels, sample containers, data sheets, and groundwater level measuring devices shall be provided in accordance with the requirements of the project and other ASTM standards.

6. Drilling Procedure

6.1 The borehole shall be advanced incrementally to permit intermittent or continuous sampling. Test intervals and locations are normally stipulated by the project engineer or geologist. Typically, the intervals selected are 5 ft (1.5 m) or less in homogeneous strata with test and sampling locations at every change of strata. Record the depth of drilling to the nearest 0.1 ft (0.030 m).

6.2 Any drilling procedure that provides a suitably clean and stable borehole before insertion of the sampler and assures that the penetration test is performed on essentially undisturbed soil shall be acceptable. Each of the following procedures has proven to be acceptable for some subsurface conditions. The subsurface conditions anticipated should be considered when selecting the drilling method to be used.

- 6.2.1 Open-hole rotary drilling method.
- 6.2.2 Continuous flight hollow-stem auger method.
- 6.2.3 Wash boring method.
- 6.2.4 Continuous flight solid auger method.

6.3 Several drilling methods produce unacceptable boreholes. The process of jetting through an open tube sampler and

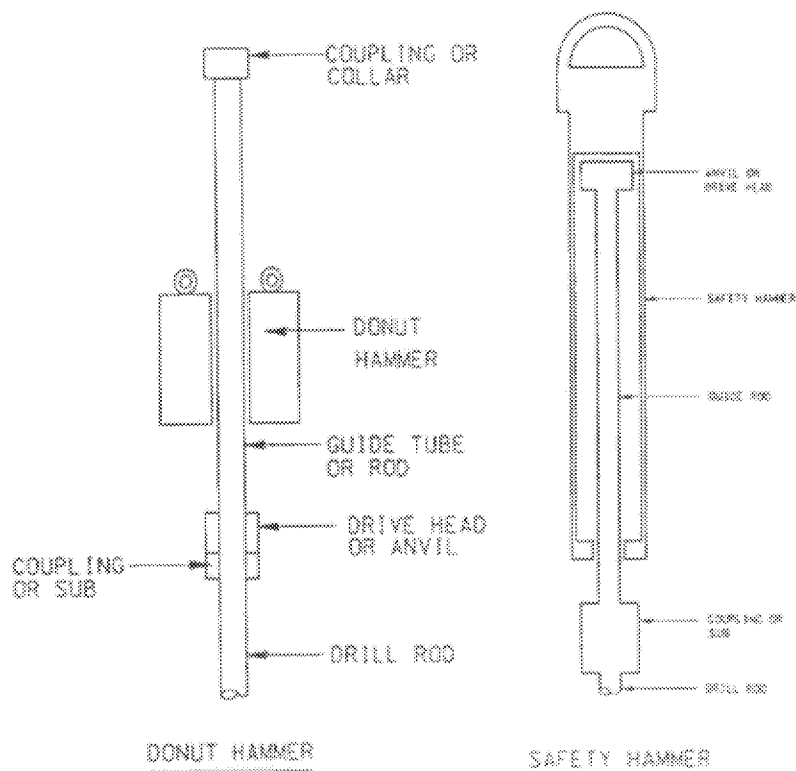


FIG. 3 Schematic Drawing of the Donut Hammer and Safety Hammer

then sampling when the desired depth is reached shall not be permitted. The continuous flight solid auger method shall not be used for advancing the borehole below a water table or below the upper confining bed of a confined non-cohesive stratum that is under artesian pressure. Casing may not be advanced below the sampling elevation prior to sampling. Advancing a borehole with bottom discharge bits is not permissible. It is not permissible to advance the borehole for subsequent insertion of the sampler solely by means of previous sampling with the SPT sampler.

6.4 The drilling fluid level within the borehole or hollow-stem augers shall be maintained at or above the in situ groundwater level at all times during drilling, removal of drill rods, and sampling.

7. Sampling and Testing Procedure

7.1 After the borehole has been advanced to the desired sampling elevation and excessive cuttings have been removed, record the cleanout depth to the nearest 0.1 ft (0.030 m), and prepare for the test with the following sequence of operations:

7.1.1 Attach either split-barrel sampler Type A or B to the sampling rods and lower into the borehole. Do not allow the sampler to drop onto the soil to be sampled.

7.1.2 Position the hammer above and attach the anvil to the top of the sampling rods. This may be done before the sampling rods and sampler are lowered into the borehole.

7.1.3 Rest the dead weight of the sampler, rods, anvil, and drive weight on the bottom of the borehole. Record the sampling start depth to the nearest 0.1 ft (0.030 m). Compare

the sampling start depth to the cleanout depth in 7.1. If excessive cuttings are encountered at the bottom of the borehole, remove the sampler and sampling rods from the borehole and remove the cuttings.

7.1.4 Mark the drill rods in three successive 0.5-foot (0.15 m) increments so that the advance of the sampler under the impact of the hammer can be easily observed for each 0.5-foot (0.15 m) increment.

7.2 Drive the sampler with blows from the 140-lbf (623-N) hammer and count the number of blows applied in each 0.5-foot (0.15-m) increment until one of the following occurs:

7.2.1 A total of 50 blows have been applied during any one of the three 0.5-foot (0.15-m) increments described in 7.1.4.

7.2.2 A total of 100 blows have been applied.

7.2.3 There is no observed advance of the sampler during the application of 10 successive blows of the hammer.

7.2.4 The sampler is advanced the complete 1.5 ft (0.45 m) without the limiting blow counts occurring as described in 7.2.1, 7.2.2, or 7.2.3.

7.2.5 If the sampler sinks under the weight of the hammer, weight of rods, or both, record the length of travel to the nearest 0.1 ft (0.030 m), and drive the sampler through the remainder of the test interval. If the sampler sinks the complete interval, stop the penetration, remove the sampler and sampling rods from the borehole, and advance the borehole through the very soft or very loose materials to the next desired sampling elevation. Record the *N*-value as either weight of hammer, weight of rods, or both.

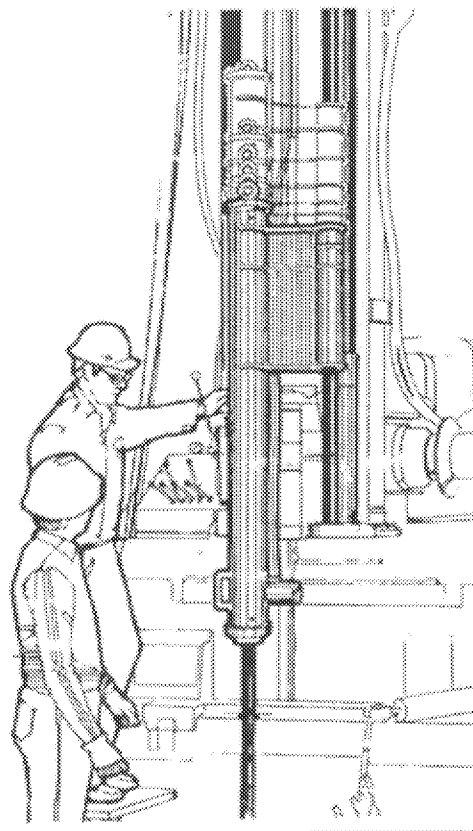


FIG. 4 Automatic Trip Hammer

7.3 Record the number of blows (N) required to advance the sampler each 0.5-foot (0.15 m) of penetration or fraction thereof. The first 0.5-foot (0.15 m) is considered to be a seating drive. The sum of the number of blows required for the second and third 0.5-foot (0.15 m) of penetration is termed the "standard penetration resistance," or the " N -value." If the sampler is driven less than 1.5 ft (0.45 m), as permitted in 7.2.1, 7.2.2, or 7.2.3, the number of blows per each complete 0.5-foot (0.15 m) increment and per each partial increment shall be recorded on the boring log. For partial increments, the depth of penetration shall be reported to the nearest 0.1 ft (0.030 m) in addition to the number of blows. If the sampler advances below the bottom of the borehole under the static weight of the drill rods or the weight of the drill rods plus the static weight of the hammer, this information should be noted on the boring log.

7.4 The raising and dropping of the 140-lbf (623-N) hammer shall be accomplished using either of the following two methods. Energy delivered to the drill rod by either method can be measured according to procedures in Test Method D 4633.

7.4.1 *Method A*—By using a trip, automatic, or semi-automatic hammer drop system that lifts the 140-lbf (623-N) hammer and allows it to drop 30 ± 1.0 in. (0.76 m \pm 0.030 m) with limited unimpedence. Drop heights adjustments for automatic and trip hammers should be checked daily and at first indication of variations in performance. Operation of automatic hammers shall be in strict accordance with operations manuals.

7.4.2 *Method B*—By using a cathead to pull a rope attached to the hammer. When the cathead and rope method is used the system and operation shall conform to the following:

7.4.2.1 The cathead shall be essentially free of rust, oil, or grease and have a diameter in the range of 6 to 10 in. (150 to 250 mm).

7.4.2.2 The cathead should be operated at a minimum speed of rotation of 100 RPM.

7.4.2.3 The operator should generally use either 1-3/4 or 2-1/4 rope turns on the cathead, depending upon whether or not the rope comes off the top (1-3/4 turns for counterclockwise rotation) or the bottom (2-1/4 turns for clockwise rotation) of the cathead during the performance of the penetration test, as shown in Fig. 1. It is generally known and accepted that 2-3/4 or more rope turns considerably impedes the fall of the hammer and should not be used to perform the test. The cathead rope should be stiff, relatively dry, clean, and should be replaced when it becomes excessively frayed, oily, limp, or burned.

7.4.2.4 For each hammer blow, a 30 ± 1.0 in. (0.76 m \pm 0.030 m) lift and drop shall be employed by the operator. The operation of pulling and throwing the rope shall be performed rhythmically without holding the rope at the top of the stroke.

Note 4—If the hammer drop height is something other than 30 ± 1.0 in. (0.76 m \pm 0.030 m), then record the new drop height. For soils other than sands, there is no known data or research that relates to adjusting the N -value obtained from different drop heights. Test method D 4633 provides information on making energy measurement for variable drop

heights and Practice D 6966 provides information on adjustment of N -value to a constant energy level (60 % of theoretical, N_{60}). Practice D 6966 allows the hammer drop height to be adjusted to provide 60 % energy.

7.5 Bring the sampler to the surface and open. Record the percent recovery to the nearest 1 % or the length of sample recovered to the nearest 0.01 ft (5 mm). Classify the soil samples recovered as to, in accordance with Practice D 2488, then place one or more representative portions of the sample into sealable moisture-proof containers (jars) without ramming or distorting any apparent stratification. Seal each container to prevent evaporation of soil moisture. Affix labels to the containers bearing job designation, boring number, sample depth, and the blow count per 0.5-foot (0.15-m) increment. Protect the samples against extreme temperature changes. If there is a soil change within the sampler, make a jar for each stratum and note its location in the sampler barrel. Samples should be preserved and transported in accordance with Practice D 4220 using Group B.

8. Data Sheet(s)/Form(s)

8.1 Data obtained in each borehole shall be recorded in accordance with the Subsurface Logging Guide D 5434 as required by the exploration program. An example of a sample data sheet is included in Appendix X1.

8.2 Drilling information shall be recorded in the field and shall include the following:

- 8.2.1 Name and location of job.
- 8.2.2 Names of crew.
- 8.2.3 Type and make of drilling machine.
- 8.2.4 Weather conditions.
- 8.2.5 Date and time of start and finish of borehole.
- 8.2.6 Boring number and location (station and coordinates, if available and applicable).
- 8.2.7 Surface elevation, if available.
- 8.2.8 Method of advancing and cleaning the borehole.
- 8.2.9 Method of keeping borehole open.
- 8.2.10 Depth of water surface to the nearest 0.1 ft (0.030 m) and drilling depth to the nearest 0.1 ft (0.030 m) at the time of a noted loss of drilling fluid, and time and date when reading or notation was made.
- 8.2.11 Location of strata changes, to the nearest 0.5 ft (15 cm).
- 8.2.12 Size of casing, depth of cased portion of borehole to the nearest 0.1 ft (0.030 m).

- 8.2.13 Equipment and Method A or B of driving sampler.
- 8.2.14 Sampler length and inside diameter of barrel, and if a sample basket retainer is used.
- 8.2.15 Size, type, and section length of the sampling rods, and
- 8.2.16 Remarks.

8.3 Data obtained for each sample shall be recorded in the field and shall include the following:

- 8.3.1 Top of sample depth to the nearest 0.1 ft (0.030 m) and, if utilized, the sample number.
- 8.3.2 Description of soil.
- 8.3.3 Strata changes within sample.
- 8.3.4 Sampler penetration and recovery lengths to the nearest 0.1 ft (0.030 m), and
- 8.3.5 Number of blows per 0.5 foot (0.15 m) or partial increment.

9. Precision and Bias

9.1 *Precision*—Test data on precision is not presented due to the nature of this test method. It is either not feasible or too costly at this time to have ten or more agencies participate in an in situ testing program at a given site.

9.1.1 The Subcommittee 18.02 is seeking additional data from the users of this test method that might be used to make a limited statement on precision. Present knowledge indicates the following:

9.1.1.1 Variations in N -values of 100 % or more have been observed when using different standard penetration test apparatus and drillers for adjacent boreholes in the same soil formation. Current opinion, based on field experience, indicates that when using the same apparatus and driller, N -values in the same soil can be reproduced with a coefficient of variation of about 10 %.

9.1.1.2 The use of faulty equipment, such as an extremely massive or damaged anvil, a rusty cathead, a low speed cathead, an old, oily rope, or massive or poorly lubricated rope sheaves can significantly contribute to differences in N -values obtained between operator-drill rig systems.

9.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

10. Keywords

10.1 blow count; in-situ test; penetration resistance; soil; split-barrel sampling; standard penetration test

APPENDIX

(Nonmandatory Information)

XI. Example Data Sheet

X1.1 See Fig. 5.

FIG. 5 Example Data Sheet



SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D 1586 – 99) that may impact the use of this standard. (Approved February 1, 2008.)

- | | |
|--|--|
| <p>(1) There have been numerous changes to this standard to list them separately. From the most recent main ballot process, additional changes were requested and incorporated into this newest revision. Stated below is a highlight of some of the changes.</p> <p>(2) Scope was completely revised.</p> <p>(3) Referenced Documents updated to include new standards.</p> | <p>(4) Terminology: added section on Definitions.</p> <p>(5) Significance and Use: clarified use of the SPT test.</p> <p>(6) Apparatus: general editorial changes.</p> <p>(7) Sampling and Testing Procedure: general editorial changes.</p> <p>(8) Data Sheets/Forms: general editorial changes.</p> <p>(9) Precision and Bias: added Sections 9.1.1.1 and 9.1.1.2.</p> |
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Designation: D 1587 – 00 (Reapproved 2007)^{e1}

Standard Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes¹

This standard is issued under the fixed designation D 1587; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

^{e1} Note—Editorial changes were made in June 2007.

1. Scope*

1.1 This practice covers a procedure for using a thin-walled metal tube to recover relatively undisturbed soil samples suitable for laboratory tests of engineering properties, such as strength, compressibility, permeability, and density. Thin-walled tubes used in piston, plug, or rotary-type samplers should comply with Section 6.3 of this practice which describes the thin-walled tubes.

Note 1—This practice does not apply to liners used within the samplers.

1.2 This Practice is limited to soils that can be penetrated by the thin-walled tube. This sampling method is not recommended for sampling soils containing gravel or larger size soil particles cemented or very hard soils. Other soil samplers may be used for sampling these soil types. Such samplers include driven split barrel samplers and soil coring devices (D 1586, D 3550, and D 6151). For information on appropriate use of other soil samplers refer to D 6169.

1.3 This practice is often used in conjunction with fluid rotary drilling (D 1452, D 5783) or hollow-stem augers (D 6151). Subsurface geotechnical explorations should be reported in accordance with practice (D 5434). This practice discusses some aspects of sample preservation after the sampling event. For information on preservation and transportation process of soil samples, consult Practice D 4230. This practice does not address environmental sampling; consult D 6169 and D 6232 for information on sampling for environmental investigations.

1.4 The values stated in inch-pound units are to be regarded as the standard. The SI values given in parentheses are provided for information purposes only. The tubing tolerances presented in Table 1 are from sources available in North

America. Use of metric equivalent is acceptable as long as thickness and proportions are similar to those required in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.6 This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.

2. Referenced Documents

2.1 ASTM Standards:²

- D 653 Terminology Relating to Soil, Rock, and Contained Fluids
- D 1452 Practice for Soil Investigation and Sampling by Auger Borings
- D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils
- D 2488 Practice for Description and Identification of Soils (Visual-Manual Procedure)
- D 3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils
- D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and Rock

¹ This practice is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Evaluations.

Current edition approved May 1, 2007. Published July 2007. Originally approved in 1958. Last previous edition approved in 2003 as D 1587 – 03.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard.

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TABLE 1 Dimensional Tolerances for Thin-Walled Tubes

| Nominal Tube Diameters from Table 2 ^a Tolerances | | | | | | |
|---|------------------|------------------|------------------|------------------|------------------|------------------|
| Size Outside Diameter | 2 | 50.8 | 3 | 76.2 | 5 | 127 |
| in. | mm | in. | mm | in. | mm | mm |
| Outside diameter, D_o | +0.007 -0.000 | +0.179 -0.000 | +0.010 -0.000 | +0.254 -0.000 | +0.015 -0.000 | 0.381 -0.000 |
| Inside diameter, D_i | +0.000 -0.007 | +0.000 -0.179 | +0.000 -0.010 | +0.000 -0.254 | +0.000 -0.015 | +0.000 -0.381 |
| Wall thickness | ±0.007 | ±0.179 | ±0.010 | ±0.254 | ±0.015 | ±0.381 |
| Roundness | 0.015 | 0.381 | 0.020 | 0.508 | 0.030 | 0.762 |
| Straightness | 0.000/ft | 2.50/m | 0.000/ft | 2.50/m | 0.000/ft | 2.50/m |

^a Intermediate or larger diameters should be proportional. Specify only two of the first three tolerances; that is, D_o and D_i , or D_o and Wall thickness, or D_i and Wall thickness.

as Used in Engineering Design and Construction

D 4220 Practices for Preserving and Transporting Soil Samples

D 5434 Guide for Field Logging of Subsurface Explorations of Soil and Rock

D 5783 Guide for Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices

D 6151 Practice for Using Hollow-Stem Augers for Geotechnical Exploration and Soil Sampling

D 6169 Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations

D 6232 Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities

3. Terminology

3.1 Definitions:

3.1.1 For common definitions of terms in this standard, refer to Terminology D 053.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *inside clearance ratio*, %, n —the ratio of the difference in the inside diameter of the tube, D_i , minus the inside diameter of the cutting edge, D_c , to the inside diameter of the tube, D_i , expressed as a percentage (see Fig. 1).

3.2.2 *ovality*, n —the cross section of the tube that deviates from a perfect circle.

4. Summary of Practice

4.1 A relatively undisturbed sample is obtained by pressing a thin-walled metal tube into the in-situ soil at the bottom of a boring, removing the soil-filled tube, and applying seals to the soil surfaces to prevent soil movement and moisture gain or loss.

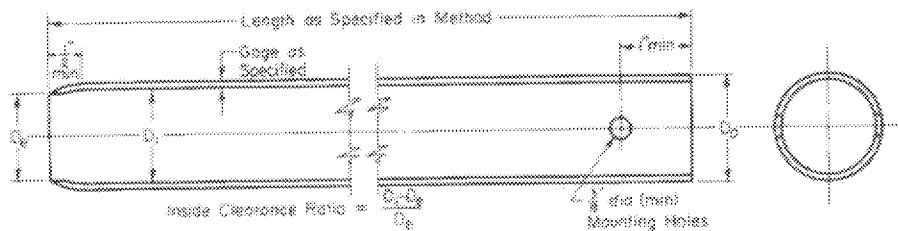
5. Significance and Use

5.1 This practice, or Practice D 3550 with thin wall shoe, is used when it is necessary to obtain a relatively undisturbed specimen suitable for laboratory tests of engineering properties or other tests that might be influenced by soil disturbance.

Note 2—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective sampling. Users of this practice, are cautioned that compliance with Practice D 3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D 3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Drilling Equipment*—When sampling in a boring, any drilling equipment may be used that provides a reasonably



Note 1—Minimum of two mounting holes on opposite sides for D_o smaller than 4 in. (101.6 mm).

Note 2—Minimum of four mounting holes equally spaced for D_o 4 in. (101.6 mm) and larger.

Note 3—Tube held with hardened screws or other suitable means.

Note 4—2-in. (50.8 mm) outside-diameter tubes are specified with an 18-gage wall thickness to comply with area ratio criteria accepted for "undisturbed samples." Users are advised that such tubing is difficult to locate and can be extremely expensive in small quantities. Sixteen-gage tubes are generally readily available.

Metric Equivalent Conversions

| | in. | mm |
|--|-----|-------|
| | 3/8 | 9.53 |
| | 1/2 | 12.7 |
| | 1 | 25.4 |
| | 2 | 50.8 |
| | 3 | 76.2 |
| | 4 | 101.6 |
| | 5 | 127 |

FIG. 1 Thin-Walled Tube for Sampling

TABLE 2 Suitable Thin-Walled Steel Sample Tubes^a

| | | | |
|-----------------------------|-------|-------|-------|
| Outside diameter (D_o): | | | |
| in. | 2 | 3 | 5 |
| mm | 50.8 | 76.2 | 127 |
| Wall thickness: | | | |
| 8wg | 18 | 18 | 11 |
| in. | 0.049 | 0.065 | 0.120 |
| mm | 1.24 | 1.65 | 3.05 |
| Tube length: | | | |
| in. | 36 | 36 | 54 |
| m | 0.91 | 0.91 | 1.45 |
| Inside clearance ratio, % | | | |
| | <1 | <1 | <1 |

^a The three diameters recommended in Table 2 are indicated for purposes of standardization, and are not intended to indicate that sampling tubes of intermediate or larger diameters are not acceptable. Lengths of tubes shown are illustrative. Proper lengths to be determined as suited to field conditions.

clean hole; that minimizes disturbance of the soil to be sampled; and that does not hinder the penetration of the thin-walled sampler. Open borehole diameter and the inside diameter of driven casing or hollow stem auger shall not exceed 3.5 times the outside diameter of the thin-walled tube.

6.2 *Sampler Insertion Equipment*, shall be adequate to provide a relatively rapid continuous penetration force. For hard formations it may be necessary, although not recommended, to drive the thin-walled tube sampler.

6.3 *Thin-Walled Tubes*, should be manufactured to the dimensions as shown in Fig. 1. They should have an outside diameter of 2 to 5 in. (50 to 130 mm) and be made of metal having adequate strength for the type of soil to be sampled. Tubes shall be clean and free of all surface irregularities including projecting weld seams. Other diameters may be used but the tube dimensions should be proportional to the tube designs presented here.

6.3.1 *Length of Tubes*—See Table 2 and 7.4.1.

6.3.2 *Tolerances*, shall be within the limits shown in Table 1.

6.3.3 *Inside Clearance Ratio*, should be not greater than 1 % unless specified otherwise for the type of soil to be sampled. Generally, the inside clearance ratio used should increase with the increase in plasticity of the soil being sampled, except for sensitive soils or where local experience indicates otherwise. See 3.2.1 and Fig. 1 for definition of inside clearance ratio.

6.3.4 *Corrosion Protection*—Corrosion, whether from galvanic or chemical reaction, can damage or destroy both the thin-walled tube and the sample. Severity of damage is a function of time as well as interaction between the sample and the tube. Thin-walled tubes should have some form of protective coating, unless the soil is to be extruded less than 3 days. The type of coating to be used may vary depending upon the material to be sampled. Plating of the tubes or alternate base metals may be specified. Galvanized tubes are often used when long term storage is required. Coatings may include a light coat of lubricating oil, lacquer, epoxy, Teflon, zinc oxide, and others.

Note 3—Most coating materials are not resistant to scratching by soils that contain sands. Consideration should be given for prompt testing of the sample because chemical reactions between the metal and the soil sample can occur with time.

6.4 *Sampler Head*, serves to couple the thin-walled tube to the insertion equipment and, together with the thin-walled tube,

comprises the thin-walled tube sampler. The sampler head shall contain a venting area and suitable check valve with the venting area to the outside equal to or greater than the area through the check valve. In some special cases, a check valve may not be required but venting is required to avoid sample compression. Attachment of the head to the tube shall be concentric and coaxial to assure uniform application of force to the tube by the sampler insertion equipment.

7. Procedure

7.1 Remove loose material from the center of a casing or hollow stem auger as carefully as possible to avoid disturbance of the material to be sampled. If groundwater is encountered, maintain the liquid level in the borehole at or above ground water level during the drilling and sampling operation.

7.2 Bottom discharge bits are not permitted. Side discharge bits may be used, with caution. Jetting through an open-tube sampler to clean out the borehole to sampling elevation is not permitted.

Note 4—Roller bits are available in downward-jetting and diffused-jet configurations. Downward-jetting configuration rock bits are not acceptable. Diffuse-jet configurations are generally acceptable.

7.3 Lower the sampling apparatus so that the sample tube's bottom rests on the bottom of the hole and record depth to the bottom of the sample tube to the nearest 0.1-ft (0.3 m).

7.3.1 Keep the sampling apparatus plumb during lowering, thereby preventing the cutting edge of the tube from scraping the wall of the borehole.

7.4^a Advance the sampler without rotation by a continuous relatively rapid downward motion and record length of advancement to the nearest 1 in. (25 mm).

7.4.1 Determine the length of advance by the resistance and condition of the soil formation, but the length shall never exceed 5 to 10 diameters of the tube in sands and 10 to 15 diameters of the tube in clays. In no case shall a length of advance be greater than the sample-tube length minus an allowance for the sampler head and a minimum of 3-in. (75 mm) for sludge and end cuttings.

Note 5—The mass of sample, laboratory handling capabilities, transportation problems, and commercial availability of tubes will generally limit maximum practical lengths to those shown in Table 2.

7.5 When the soil formation is too hard for push-type insertion, the tube may be driven or Practice D 3550 may be used. If driving methods are used, the data regarding weight and fall of the hammer and penetration achieved must be shown in the report. Additionally, that tube must be prominently labeled a "driven sample."

7.6 Withdraw the sampler from the soil formation as carefully as possible in order to minimize disturbance of the sample. The tube can be slowly rotated to shear the material at the end of the tube, and to relieve water and/or suction pressures and improve recovery. Where the soil formation is soft, a delay before withdraw of the sampler (typically 5 to 30 minutes) may improve sample recovery.

8. Sample Measurement, Sealing and Labeling

8.1 Upon removal of the tube, remove the drill cuttings in the upper end of the tube and measure the length of the soil

sample recovered to the nearest 0.25 in. (5 mm) in the tube. Seal the upper end of the tube. Remove at least 1 in. (25 mm) of material from the lower end of the tube. Use this material for soil description in accordance with Practice D 2488. Measure the overall sample length. Seal the lower end of the tube. Alternatively, after measurement, the tube may be sealed without removal of soil from the ends of the tube.

8.1.1 Tubes sealed over the ends, as opposed to those sealed with expanding packers, should be provided with spacers or appropriate packing materials, or both prior to sealing the tube ends to provide proper confinement. Packing materials must be nonabsorbent and must maintain their properties to provide the same degree of sample support with time.

8.1.2 Depending on the requirements of the investigation, field extrusion and packaging of extruded soil samples can be performed. This allows for physical examination and classification of the sample. Samples are extruded in special hydraulic jacks equipped with properly sized platens to extrude the core in a continuous smooth speed. In some cases, further extrusion may cause sample disturbance reducing suitability for testing of engineering properties. In other cases, if damage is not significant, cores can be extruded and preserved for testing (D-42.26). Bent or damaged tubes should be cut off before extruding.

8.2 Prepare and immediately affix labels or apply markings as necessary to identify the sample (see Section 9). Assure that the markings or labels are adequate to survive transportation and storage.

Note 6—Top end of the tube should be labeled "top"

9. Final Log

9.1 Record the information that may be required for preparing field logs in general accordance to ASTM D 5434 "Guide

for Field Logging of Subsurface Explorations of Soil and Rock". This guide is used for logging explorations by drilling and sampling. Some examples of the information required include:

- 9.1.1 Name and location of the project,
- 9.1.2 Boring number,
- 9.1.3 Log of the soil conditions,
- 9.1.4 Surface elevation or reference to a datum to the nearest foot (0.5 m) or better,
- 9.1.5 Location of the boring,
- 9.1.6 Method of making the borehole,
- 9.1.7 Name of the drilling foreman and company, and
- 9.1.8 Name of the drilling inspector(s),
- 9.1.9 Date and time of boring-start and finish,
- 9.1.10 Depth to groundwater level; date and time measured,
- 9.2 Recording the appropriate sampling information is required as follows:
 - 9.2.1 Depth to top of sample to the nearest 0.1 ft. (0.3 m) and number of sample,
 - 9.2.2 Description of thin-walled tube sampler: size, type of metal, type of coating,
 - 9.2.3 Method of sampler insertion: push or drive,
 - 9.2.4 Method of drilling, size of hole, casing, and drilling fluid used,
 - 9.2.5 Soil description in accordance with Practice D 2488,
 - 9.2.6 Length of sampler advance (push), and
 - 9.2.7 Recovery: length of sample obtained,

10. Keywords

10.1 geologic investigations; sampling; soil exploration;
soil investigations; subsurface investigations; undisturbed

SUMMARY OF CHANGES

In accordance with committee D18 policy, this section identifies the location of changes to this standard since the last edition, 200, which may impact the use of this standard.

- (1) Added parts of speech to terms. (2) Corrected reference in Note 2 from D 5740 to D 5741.

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Logging of Soil Borings

I. Purpose and Scope

This SOP provides guidance to obtain accurate and consistent descriptions of soil characteristics during soil-sampling operations. The characterization is based on visual examination and manual tests, not on laboratory determinations.

II. Equipment and Materials

- Indelible pens
- Tape measure or ruler
- Field logbook
- Spatula
- HCL, 10 percent solution
- Squirt bottle with water
- Rock- or soil-color chart (e.g., Munsell)
- Grain-size chart
- Hand lens
- Unified Soil Classification System (USCS) index charts and tables to help with soil classification (attached)

III. Procedures and Guidelines

This section covers several aspects of soil characterization: instructions for completing the CH2M soil boring log Form D1586 (attached), field classification of soil, and standard penetration test procedures.

A. Instructions for Completing Soil Boring Logs

Soil boring logs will be completed in the field log books or on separate soil boring log sheets. Information collected will be consistent with that required for Form D1586 (attached), a standard CH2M form (attached), or an equivalent form that supplies the same information.

The information collected in the field to perform the soil characterization is described below.

Field personnel should review completed logs for accuracy, clarity, and thoroughness of detail. Samples also should be checked to see that information is correctly recorded on both jar lids and labels and on the log sheets.

B. Heading Information

Boring/Well Number. Enter the boring/well number. A numbering system should be chosen that does not conflict with information recorded for previous exploratory work done at the site. Number the sheets consecutively for each boring.

Location. If station, coordinates, mileposts, or similar project layout information is available, indicate the position of the boring to that system using modifiers such as "approximate" or "estimated" as appropriate.

Elevation. Elevation will be determined at the conclusion of field activities through a survey.

Drilling Contractor. Enter the name of the drilling company and the city and state where the company is based.

Drilling Method and Equipment. Identify the bit size and type, drilling fluid (if used), and method of drilling (e.g., rotary, hollow-stem auger, sonic). Information on the drilling equipment (e.g., CME 55, Mobile B61) also is noted.

Water Level and Date. Enter the depth below ground surface to the apparent water level in the borehole. The information should be recorded as a comment. If free water is not encountered during drilling or cannot be detected because of the drilling method, this information should be noted. Record date and time of day (for tides, river stage) of each water level measurement.

Date of Start and Finish. Enter the dates the boring was begun and completed. Time of day should be added if several borings are performed on the same day.

Logger. Enter the first and last name.

C. Technical Data

Depth Below Surface. Use a depth scale that is appropriate for the sample spacing and for the complexity of subsurface conditions.

Sample Interval. Note the depth at the top and bottom of the sample interval.

Sample Type and Number. Enter the sample type and number. SS-1 = split spoon, first sample. Number samples consecutively regardless of type. Enter a sample number even if no material was recovered in the sampler.

Sample Recovery. Enter the length to the nearest 0.1-foot of soil sample recovered from the sampler. Often, there will be some wash or caved material above the sample; do not include the wash material in the measurement. Record soil recovery in feet.

Standard Penetration Test Results. In this column, enter the number of blows required for each 6 inches of sampler penetration and the "N" value, which is the sum of the blows in the middle two 6-inch penetration intervals. A typical standard penetration test involving successive blow counts of 2, 3, 4, and 5 is recorded as 2-3-4-5 and (7). The standard penetration test is terminated if the sampler encounters refusal. Refusal is a penetration of less than 6 inches with a blow count of 50. A

partial penetration of 50 blows for 4 inches is recorded as 50/4 inches. Penetration by the weight of the slide hammer only is recorded as "WOH."

Samples should be collected using a 140-pound hammer and 2-inch diameter split spoons. Samples may be collected using direct push sampling equipment. However, blow counts will not be available. A pocket penetrometer may be used instead to determine relative soil density of fine grained materials (silts and clays).

Sample also may be collected using a 300-pound hammer or 3-inch-diameter split-spoon samples at the site. However, use of either of these sample collection devices invalidates standard penetration test results and should be noted in the comments section of the log. The 300-pound hammer should only be used for collection of 3-inch-diameter split-spoon samples. Blow counts should be recorded for collection of samples using either a 3-inch split-spoon, or a 300-pound hammer. An "N" value need not be calculated.

Soil Description. The soil classification should follow the format described in the "Field Classification of Soil" subsection below.

Comments. Include all pertinent observations (changes in drilling fluid color, rod drops, drilling chatter, rod bounce as in driving on a cobble, damaged Shelby tubes, and equipment malfunctions). In addition, note if casing was used, the sizes and depths installed, and if drilling fluid was added or changed. You should instruct the driller to alert you to any significant changes in drilling (changes in material, occurrence of boulders, and loss of drilling fluid). Such information should be attributed to the driller and recorded in this column.

Specific information might include the following:

- The date and the time drilling began and ended each day
- The depth and size of casing and the method of installation
- The date, time, and depth of water level measurements
- Depth of rod chatter
- Depth and percentage of drilling fluid loss
- Depth of hole caving or heaving
- Depth of change in material
- Health and safety monitoring data
- Drilling interval through a boulder

D. Field Classification of Soil

This section presents the format for the field classification of soil. In general, the approach and format for classifying soils should conform to ASTM D 2488, Visual-Manual Procedure for Description and Identification of Soils (attached).

The Unified Soil Classification System is based on numerical values of certain soil properties that are measured by laboratory tests. It is possible, however, to estimate these values in the field with reasonable accuracy using visual-manual procedures (ASTM D 2488). In addition, some elements of a complete soil

description, such as the presence of cobbles or boulders, changes in strata, and the relative proportions of soil types in a bedded deposit, can be obtained only in the field.

Soil descriptions should be precise and comprehensive without being verbose. The correct overall impression of the soil should not be distorted by excessive emphasis on insignificant details. In general, similarities rather than differences between consecutive samples should be stressed.

Soil descriptions must be recorded for every soil sample collected. The format and order for soil descriptions should be as follows:

1. Soil name (synonymous with ASTM D 2488 Group Name) with appropriate modifiers. Soil name should be in all capitals in the log, for example "POORLY-GRADED SAND."
2. Group symbol, in parentheses, for example, "(SP)."
3. Color, using Munsell color designation
4. Moisture content
5. Relative density or consistency
6. Soil structure, mineralogy, or other descriptors

This order follows, in general, the format described in ASTM D 2488.

E. Soil Name

The basic name of a soil should be the ASTM D 2488 Group Name on the basis of visual estimates of gradation and plasticity. The soil name should be capitalized.

Examples of acceptable soil names are illustrated by the following descriptions:

- A soil sample is visually estimated to contain 15 percent gravel, 55 percent sand, and 30 percent fines (passing No. 200 sieve). The fines are estimated as either low or highly plastic silt. This visual classification is SILTY SAND WITH GRAVEL, with a Group Symbol of (SM).
- Another soil sample has the following visual estimate: 10 percent gravel, 30 percent sand, and 60 percent fines (passing the No. 200 sieve). The fines are estimated as low plastic silt. This visual classification is SANDY SILT. The gravel portion is not included in the soil name because the gravel portion was estimated as less than 15 percent. The Group Symbol is (ML).

The gradation of coarse-grained soil (more than 50 percent retained on No. 200 sieve) is included in the specific soil name in accordance with ASTM D 2488. There is no need to further document the gradation. However, the maximum size and angularity or roundness of gravel and sand-sized particles should be recorded. For fine-grained soil (50 percent or more passing the No. 200 sieve), the name is modified by the appropriate plasticity/elasticity term in accordance with ASTM D 2488.

Interlayered soil should each be described starting with the predominant type. An introductory name, such as “Interlayered Sand and Silt,” should be used. In addition, the relative proportion of each soil type should be indicated (see Table 1 for example).

Where helpful, the evaluation of plasticity/elasticity can be justified by describing results from any of the visual-manual procedures for identifying fine-grained soils, such as reaction to shaking, toughness of a soil thread, or dry strength as described in ASTM D 2488.

F. Group Symbol

The appropriate group symbol from ASTM D 2488 must be given after each soil name. The group symbol should be placed in parentheses to indicate that the classification has been estimated.

In accordance with ASTM D 2488, dual symbols (e.g., GP-GM or SW-SC) can be used to indicate that a soil is estimated to have about 10 percent fines. Borderline symbols (e.g., GM/SM or SW/SP) can be used to indicate that a soil sample has been identified as having properties that do not distinctly place the soil into a specific group. Generally, the group name assigned to a soil with a borderline symbol should be the group name for the first symbol. The use of a borderline symbol should not be used indiscriminately. Every effort should be made to first place the soil into a single group.

G. Color

The color of a soil must be given. The color description should be based on the Munsell system. The color name and the hue, value, and chroma should be given.

H. Moisture Content

The degree of moisture present in a soil sample should be defined as dry, moist, or wet. Moisture content can be estimated from the criteria listed on Table 2.

I. Relative Density or Consistency

Relative density of a coarse-grained (cohesionless) soil is based on N-values (ASTM D 1586 [attached]). If the presence of large gravel, disturbance of the sample, or non-standard sample collection makes determination of the in situ relative density or consistency difficult, then this item should be left out of the description and explained in the Comments column of the soil boring log.

Consistency of fine-grained (cohesive) soil is properly based on results of pocket penetrometer or torvane results. In the absence of this information, consistency can be estimated from N-values. Relationships for determining relative density or consistency of soil samples are given in Tables 3 and 4.

J. Soil Structure, Mineralogy, and Other Descriptors

Discontinuities and inclusions are important and should be described. Such features include joints or fissures, slickensides, bedding or laminations, veins, root holes, and wood debris.

Significant mineralogical information such as cementation, abundant mica, or unusual mineralogy should be described.

Other descriptors may include particle size range or percentages, particle angularity or shape, maximum particle size, hardness of large particles, plasticity of fines, dry strength, dilatancy, toughness, reaction to HCl, and staining, as well as other information such as organic debris, odor, or presence of free product.

K. Equipment and Calibration

Before starting the testing, the equipment should be inspected for compliance with the requirements of ASTM D 1586. The split-barrel sampler should measure 2-inch or 3-inch O.D., and should have a split tube at least 18 inches long. The minimum size sampler rod allowed is "A" rod (1-5/8-inch O.D.). A stiffer rod, such as an "N" rod (2-5/8-inch O.D.), is required for depths greater than 50 feet. The drive weight assembly should consist of a 140-pound or 300-pound hammer weight, a drive head, and a hammer guide that permits a free fall of 30 inches.

IV. Attachments

Soil Boring Log (Sample Soil Boring Log.xls)

CH2M Form D1586 and a completed example (Soil_Log_Examp.pdf)

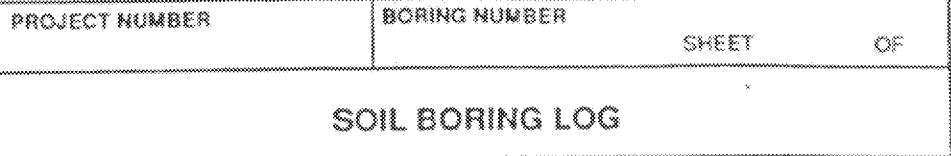
ASTM D 2488 *Standard Practice for Description and Identification of Soils (Visual-Manual Procedures)* (ASTM D2488.pdf)

ASTM 1586 *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils* (ASTM D1586.pdf)

Tables 1 through 4 (Tables 1-4.pdf)

V. Key Checks and Preventive Maintenance

- Check entries to the soil-boring log and field logbook in the field; because the samples will be disposed of at the end of fieldwork, confirmation and corrections cannot be made later.
- Check that sample numbers and intervals are properly specified.
- Check that drilling and sampling equipment is decontaminated using the procedures defined in SOP *Decontamination of Drilling Rigs and Equipment*.

[illegible]

REV : 1/86 : Q944 03586



| | | |
|---------------------------------------|------------------------------|-------------------------------|
| PROJECT NUMBER <u>DEN 22371.G5</u> | BORING NUMBER <u>BL-3</u> | SHEET <u>1</u> OF <u>3</u> |
| SOIL BORING LOG | | |

PROJECT Howard Ave Landslide LOCATION Howard Ave, Centennial, CO
ELEVATION 513 1/2 Feet DRILLING CONTRACTOR Kendall Explorations, Arden, Colorado
DRILLING METHOD AND EQUIPMENT 4" inch H.S. Augers, Mobil B-61 rotary drill rig
WATER LEVELS 3.2 Feet, 8/5/89 START August 4, 1989 FINISH August 8, 1989 LOGGER J.A. Michner

| DEPTH BELOW SURFACE (FT) | SAMPLE | | | STANDARD PENETRATION TEST RESULTS 6"-6"-6" (N) | SOIL DESCRIPTION SOIL NAME, USCS GROUP SYMBOL, COLOR, MOISTURE CONTENT, RELATIVE DENSITY OR CONSISTENCY, SOIL STRUCTURE, MINERALOGY | COMMENTS DEPTH OF CASING, DRILLING RATE, DRILLING FLUID LOSS, TESTS AND INSTRUMENTATION |
|--------------------------|----------|-----------------|---------------|--|--|---|
| | INTERVAL | NUMBER AND TYPE | RECOVERY (FT) | | | |
| 0 | | | | | Surface material consist of 4 inches AC underlain by 6 inches of 3/4 inch minus base rock | Start Drilling @ 3:00 |
| 2.5 | | | | | | |
| 4.0 | 1-S | 1.5 | | 2-3-4 (7) | POORLY-GRADED SAND WITH SILT, (SP-SM), fine, light brown, wet, loose | Driller notes water at 4 feet |
| 5.0 | | | | | | Driller notes very soft drilling |
| 6.5 | 2-S | 0.9 | | NOH/12-1 | ORGANIC SILT, (OL), very dark, gray to black, wet, very soft, strong H ₂ S odor, many fine roots up to about 1/4 inch | 4 ft. dark grey, wet silty cuttings. |
| 8.0 | | | | | | |
| 10.0 | 3-ST | 1.3 | | | ORGANIC SILT, similar to 2-S, except includes fewer roots (by volume) | |
| 11.5 | 4-S | 1.3 | | 2-2-2 (4) | SILT, (ML), very dark gray to black, wet, soft | |
| 15.0 | | | | | | water level @ 3.2 feet on 8/5/89 @ 0130 |
| 15.5 | 5-S | 0.5 | | 60/6" | SILTY GRAVEL, (GM), rounded gravel up to about 1 inch maximum observed size, wet, very dense | Driller notes rough drilling action and chatter @ 13 ft. |
| 20.0 | | | | | | |
| 21.0 | 6-S | 1.0 | | 12-50/6" | LEAN CLAY WITH SAND, (CL), medium to light green, moist, very stiff | Driller notes smoother, firm drilling @ 19 ft. some angular rock chips @ bot. of 6-S, poss boulders or rock |
| 23.0 | | | | | | Driller notes very hard, slow grinding, smooth drilling action from 21 to 23 ft, possibly bedrock |
| 23.1 | 7-S | 0 | | 50/1" | NO RECOVERY | |
| | | | | | END SOIL BORING @ 23.1 FEET SEE ROCK CORE LOG FOR CONTINUATION OF BL-3 | |

Figure 2
EXAMPLE OF COMPLETED LOG FORM



Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)¹

This standard is issued under the fixed designation D 2488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This practice covers procedures for the description of soils for engineering purposes.

1.2 This practice also describes a procedure for identifying soils, at the option of the user, based on the classification system described in Test Method D 2487. The identification is based on visual examination and manual tests. It must be clearly stated in reporting an identification that it is based on visual-manual procedures.

1.2.1 When precise classification of soils for engineering purposes is required, the procedures prescribed in Test Method D 2487 shall be used.

1.2.2 In this practice, the identification portion assigning a group symbol and name is limited to soil particles smaller than 3 in. (75 mm).

1.2.3 The identification portion of this practice is limited to naturally occurring soils (disturbed and undisturbed).

Note 1—This practice may be used as a descriptive system applied to such materials as shale, claystone, shells, crushed rock, etc. (see Appendix X2).

1.3 The descriptive information in this practice may be used with other soil classification systems or for materials other than naturally occurring soils.

1.4 The values stated in inch-pound units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements see Section 8.*

1.6 *This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which*

the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.

2. Referenced Documents

2.1 ASTM Standards:

- D 653 Terminology Relating to Soil, Rock, and Contained Fluids²
- D 1452 Practice for Soil Investigation and Sampling by Auger Borings²
- D 1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils²
- D 1587 Practice for Thin-Walled Tube Sampling of Soils²
- D 2113 Practice for Diamond Core Drilling for Site Investigation²
- D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)²
- D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction³
- D 4083 Practice for Description of Frozen Soils (Visual-Manual Procedure)²

3. Terminology

3.1 **Definitions**—Except as listed below, all definitions are in accordance with Terminology D 653.

Note 2—For particles retained on a 3-in. (75-mm) US standard sieve, the following definitions are suggested:

Cobbles—particles of rock that will pass a 12-in. (300-mm) square opening and be retained on a 3-in. (75-mm) sieve, and
Boulders—particles of rock that will not pass a 12-in. (300-mm) square opening

3.1.1 **clay**—soil passing a No. 200 (75- μ m) sieve that can be made to exhibit plasticity (putty-like properties) within a range of water contents, and that exhibits considerable strength when air-dry. For classification, a clay is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index equal to or greater than 4, and the plot of plasticity index versus liquid

¹ This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.07 on Identification and Classification of Soils.

Current edition approved Feb. 10, 2000. Published May 2000. Originally published as D 2488 – 66 T. Last previous edition D 2488 – 93*.

² *Annual Book of ASTM Standards*, Vol 04.08.

³ *Annual Book of ASTM Standards*, Vol 04.09.

*A Summary of Changes section appears at the end of this standard.

limit falls on or above the "A" line (see Fig. 3 of Test Method D 2487).

3.1.2 *gravel*—particles of rock that will pass a 3-in. (75-mm) sieve and be retained on a No. 4 (4.75-mm) sieve with the following subdivisions:

coarse—passes a 3-in. (75-mm) sieve and is retained on a ½-in. (19-mm) sieve.

fine—passes a ½-in. (19-mm) sieve and is retained on a No. 4 (4.75-mm) sieve.

3.1.3 *organic clay*—a clay with sufficient organic content to influence the soil properties. For classification, an organic clay is a soil that would be classified as a clay, except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.4 *organic silt*—a silt with sufficient organic content to influence the soil properties. For classification, an organic silt is a soil that would be classified as a silt except that its liquid limit value after oven drying is less than 75 % of its liquid limit value before oven drying.

3.1.5 *peat*—a soil composed primarily of vegetable tissue in various stages of decomposition usually with an organic odor, a dark brown to black color, a spongy consistency, and a texture ranging from fibrous to amorphous.

3.1.6 *sand*—particles of rock that will pass a No. 4 (4.75-mm) sieve and be retained on a No. 200 (75-µm) sieve with the following subdivisions:

coarse—passes a No. 4 (4.75-mm) sieve and is retained on a No. 10 (2.00-mm) sieve.

medium—passes a No. 10 (2.00-mm) sieve and is retained on a No. 40 (425-µm) sieve.

fine—passes a No. 40 (425-µm) sieve and is retained on a No. 200 (75-µm) sieve.

3.1.7 *silt*—soil passing a No. 200 (75-µm) sieve that is nonplastic or very slightly plastic and that exhibits little or no strength when air dry. For classification, a silt is a fine-grained soil, or the fine-grained portion of a soil, with a plasticity index less than 4, or the plot of plasticity index versus liquid limit falls below the "A" line (see Fig. 3 of Test Method D 2487).

4. Summary of Practice

4.1 Using visual examination and simple manual tests, this practice gives standardized criteria and procedures for describing and identifying soils.

4.2 The soil can be given an identification by assigning a group symbol(s) and name. The flow charts, Fig. 1a and Fig. 1b for fine-grained soils, and Fig. 2, for coarse-grained soils, can be used to assign the appropriate group symbol(s) and name. If the soil has properties which do not distinctly place it into a specific group, borderline symbols may be used, see Appendix X3.

Note 3—It is suggested that a distinction be made between *dual symbols* and *borderline symbols*.

Dual Symbol—A dual symbol is two symbols separated by a hyphen, for example, GP-GM, SW-SC, CL-ML, used to indicate that the soil has been identified as having the properties of a classification in accordance with Test Method D 2487 where two symbols are required. Two symbols are required when the soil has between 5 and 12 % fines or when the liquid limit and plasticity index values plot in the CL-ML area of the plasticity chart.

Borderline Symbol—A borderline symbol is two symbols separated by a slash, for example, CL/CH, GM/SM, CL/ML. A borderline symbol should be used to indicate that the soil has been identified as having properties that do not distinctly place the soil into a specific group (see Appendix X3).

5. Significance and Use

5.1 The descriptive information required in this practice can be used to describe a soil to aid in the evaluation of its significant properties for engineering use.

5.2 The descriptive information required in this practice should be used to supplement the classification of a soil as determined by Test Method D 2487.

5.3 This practice may be used in identifying soils using the classification group symbols and names as prescribed in Test Method D 2487. Since the names and symbols used in this practice to identify the soils are the same as those used in Test Method D 2487, it shall be clearly stated in reports and all other appropriate documents, that the classification symbol and name are based on visual-manual procedures.

5.4 This practice is to be used not only for identification of soils in the field, but also in the office, laboratory, or wherever soil samples are inspected and described.

5.5 This practice has particular value in grouping similar soil samples so that only a minimum number of laboratory tests need be run for positive soil classification.

Note 4—The ability to describe and identify soils correctly is learned more readily under the guidance of experienced personnel, but it may also be acquired systematically by comparing numerical laboratory test results for typical soils of each type with their visual and manual characteristics.

5.6 When describing and identifying soil samples from a given boring, test pit, or group of borings or pits, it is not necessary to follow all of the procedures in this practice for every sample. Soils which appear to be similar can be grouped together; one sample completely described and identified with the others referred to as similar based on performing only a few of the descriptive and identification procedures described in this practice.

5.7 This practice may be used in combination with Practice D 4083 when working with frozen soils.

Note 5—Notwithstanding the statements on precision and bias contained in this standard, the precision of this test method is dependent on the competence of the personnel performing it and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D 3740 does not in itself assure reliable testing. Reliable testing depends on several factors; Practice D 3740 provides a means for evaluating some of those factors.

6. Apparatus

6.1 Required Apparatus:

6.1.1 Pocket Knife or Small Spatula.

6.2 Useful Auxiliary Apparatus:

6.2.1 Small Test Tube and Stopper (or jar with a lid).

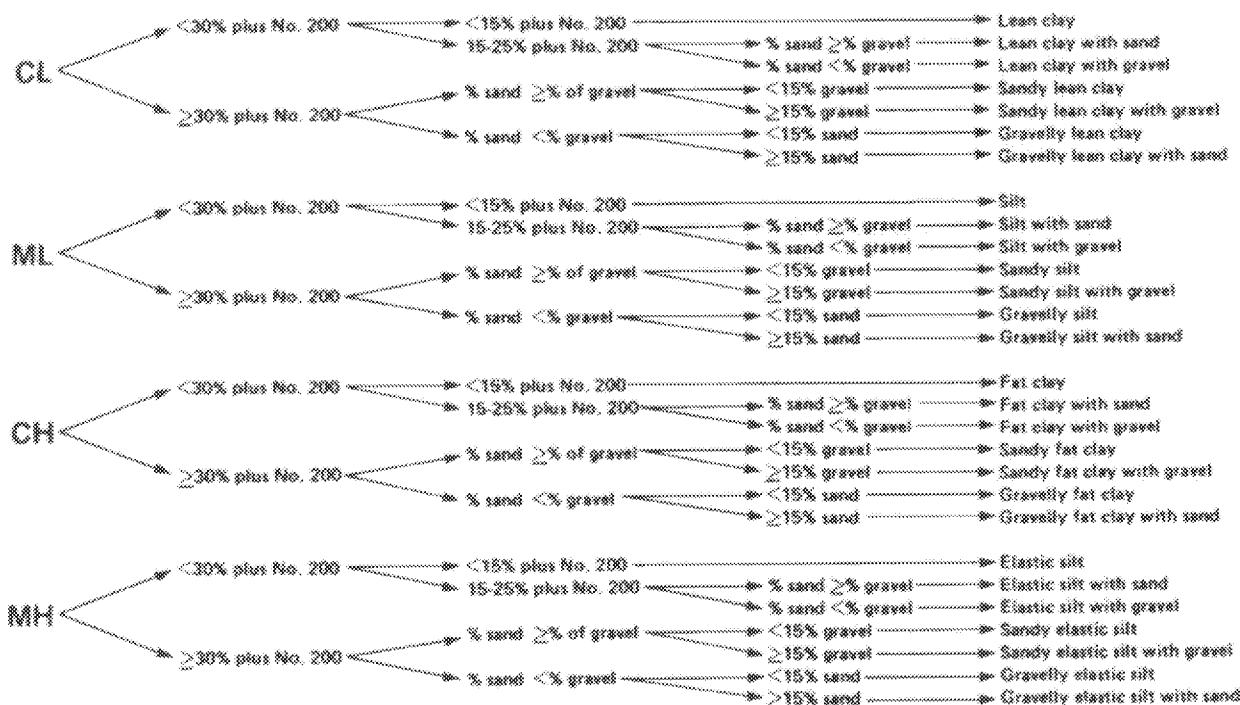
6.2.2 Small Hand Lens.

7. Reagents

7.1 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water from a city water

GROUP SYMBOL

GROUP NAME

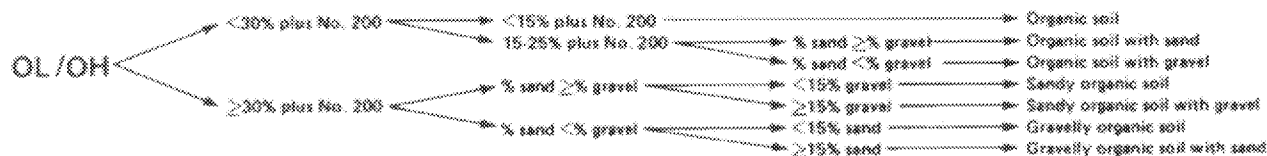


NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1a Flow Chart for Identifying Inorganic Fine-Grained Soil (50 % or more fines)

GROUP SYMBOL

GROUP NAME



NOTE 1—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 1 b Flow Chart for Identifying Organic Fine-Grained Soil (50 % or more fines)

supply or natural source, including non-potable water.

7.2 *Hydrochloric Acid*—A small bottle of dilute hydrochloric acid, HCl, one part HCl (10 N) to three parts water (This reagent is optional for use with this practice). See Section 8.

8. Safety Precautions

8.1 When preparing the dilute HCl solution of one part concentrated hydrochloric acid (10 N) to three parts of distilled water, slowly add acid into water following necessary safety precautions. Handle with caution and store safely. If solution comes into contact with the skin, rinse thoroughly with water.

8.2 **Caution**—Do not add water to acid.

9. Sampling

9.1 The sample shall be considered to be representative of the stratum from which it was obtained by an appropriate, accepted, or standard procedure.

Note 6—Preferably, the sampling procedure should be identified as

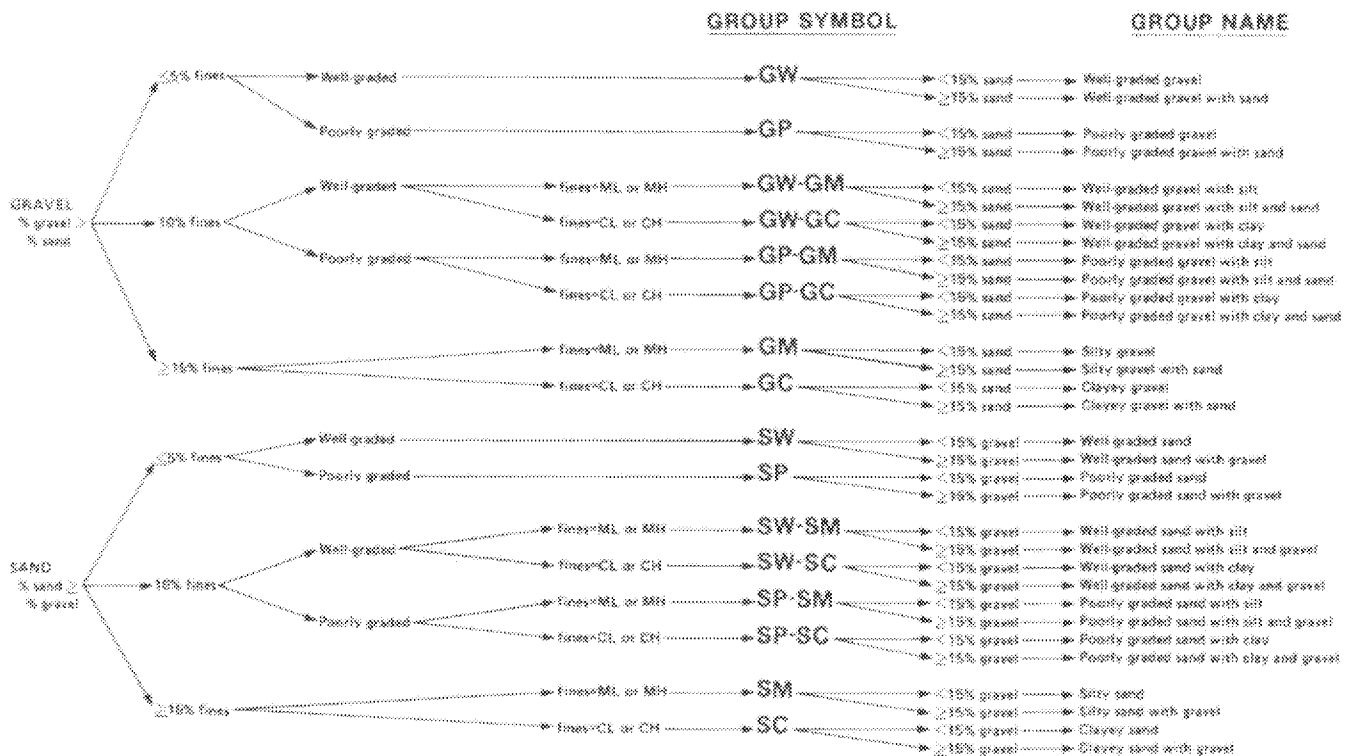
having been conducted in accordance with Practices D 1452, D 1587, or D 2113, or Test Method D 1586.

9.2 The sample shall be carefully identified as to origin.

Note 7—Remarks as to the origin may take the form of a boring number and sample number in conjunction with a job number, a geologic stratum, a pedologic horizon or a location description with respect to a permanent monument, a grid system or a station number and offset with respect to a stated centerline and a depth or elevation.

9.3 For accurate description and identification, the minimum amount of the specimen to be examined shall be in accordance with the following schedule:

| Maximum Particle Size, Sieve Opening | Minimum Specimen Size, Dry Weight |
|---|--------------------------------------|
| 4.75 mm (No. 4) | 100 g (0.25 lb) |
| 9.5 mm (3/8 in.) | 200 g (0.5 lb) |
| 19.0 mm (3/4 in.) | 1.0 kg (2.2 lb) |
| 38.1 mm (1 1/2 in.) | 6.0 kg (13 lb) |
| 75.0 mm (3 in.) | 60.0 kg (132 lb) |



Note 1.—Percentages are based on estimating amounts of fines, sand, and gravel to the nearest 5 %.

FIG. 2 Flow Chart for Identifying Coarse-Grained Soils (less than 50 % fines)

Note 8.—If random isolated particles are encountered that are significantly larger than the particles in the soil matrix, the soil matrix can be accurately described and identified in accordance with the preceding schedule.

9.4 If the field sample or specimen being examined is smaller than the minimum recommended amount, the report shall include an appropriate remark.

10. Descriptive Information for Soils

10.1 *Angularity*—Describe the angularity of the sand (coarse sizes only), gravel, cobbles, and boulders, as angular, subangular, subrounded, or rounded in accordance with the criteria in Table 1 and Fig. 3. A range of angularity may be stated, such as: subrounded to rounded.

10.2 *Shape*—Describe the shape of the gravel, cobbles, and boulders as flat, elongated, or flat and elongated if they meet the criteria in Table 2 and Fig. 4. Otherwise, do not mention the shape. Indicate the fraction of the particles that have the shape, such as: one-third of the gravel particles are flat.

TABLE 1 Criteria for Describing Angularity of Coarse-Grained Particles (see Fig. 3)

| Description | Criteria |
|-------------|--|
| Angular | Particles have sharp edges and relatively plane sides with unpolished surfaces |
| Subangular | Particles are similar to angular description but have rounded edges |
| Subrounded | Particles have nearly plane sides but have well-rounded corners and edges |
| Rounded | Particles have smoothly curved sides and no edges |

10.3 *Color*—Describe the color. Color is an important property in identifying organic soils, and within a given locality it may also be useful in identifying materials of similar geologic origin. If the sample contains layers or patches of varying colors, this shall be noted and all representative colors shall be described. The color shall be described for moist samples. If the color represents a dry condition, this shall be stated in the report.

10.4 *Odor*—Describe the odor if organic or unusual. Soils containing a significant amount of organic material usually have a distinctive odor of decaying vegetation. This is especially apparent in fresh samples, but if the samples are dried, the odor may often be revived by heating a moistened sample. If the odor is unusual (petroleum product, chemical, and the like), it shall be described.

10.5 *Moisture Condition*—Describe the moisture condition as dry, moist, or wet, in accordance with the criteria in Table 3.

10.6 *HCl Reaction*—Describe the reaction with HCl as none, weak, or strong, in accordance with the criteria in Table 4. Since calcium carbonate is a common cementing agent, a report of its presence on the basis of the reaction with dilute hydrochloric acid is important.

10.7 *Consistency*—For intact fine-grained soil, describe the consistency as very soft, soft, firm, hard, or very hard, in accordance with the criteria in Table 5. This observation is inappropriate for soils with significant amounts of gravel.

10.8 *Cementation*—Describe the cementation of intact coarse-grained soils as weak, moderate, or strong, in accordance with the criteria in Table 6.



2.2.2. *Phylogenetic analysis*

1998, 1999, 2000, 2001, 2002, 2003, 2004, 2005, 2006, 2007, 2008, 2009, 2010, 2011, 2012, 2013, 2014, 2015, 2016, 2017, 2018, 2019, 2020, 2021, 2022, 2023, 2024, 2025, 2026, 2027, 2028, 2029, 2030, 2031, 2032, 2033, 2034, 2035, 2036, 2037, 2038, 2039, 2040, 2041, 2042, 2043, 2044, 2045, 2046, 2047, 2048, 2049, 2050, 2051, 2052, 2053, 2054, 2055, 2056, 2057, 2058, 2059, 2060, 2061, 2062, 2063, 2064, 2065, 2066, 2067, 2068, 2069, 2070, 2071, 2072, 2073, 2074, 2075, 2076, 2077, 2078, 2079, 2080, 2081, 2082, 2083, 2084, 2085, 2086, 2087, 2088, 2089, 2090, 2091, 2092, 2093, 2094, 2095, 2096, 2097, 2098, 2099, 2100, 2101, 2102, 2103, 2104, 2105, 2106, 2107, 2108, 2109, 2110, 2111, 2112, 2113, 2114, 2115, 2116, 2117, 2118, 2119, 2120, 2121, 2122, 2123, 2124, 2125, 2126, 2127, 2128, 2129, 2130, 2131, 2132, 2133, 2134, 2135, 2136, 2137, 2138, 2139, 2140, 2141, 2142, 2143, 2144, 2145, 2146, 2147, 2148, 2149, 2150, 2151, 2152, 2153, 2154, 2155, 2156, 2157, 2158, 2159, 2160, 2161, 2162, 2163, 2164, 2165, 2166, 2167, 2168, 2169, 2170, 2171, 2172, 2173, 2174, 2175, 2176, 2177, 2178, 2179, 2180, 2181, 2182, 2183, 2184, 2185, 2186, 2187, 2188, 2189, 2190, 2191, 2192, 2193, 2194, 2195, 2196, 2197, 2198, 2199, 2200, 2201, 2202, 2203, 2204, 2205, 2206, 2207, 2208, 2209, 2210, 2211, 2212, 2213, 2214, 2215, 2216, 2217, 2218, 2219, 2220, 2221, 2222, 2223, 2224, 2225, 2226, 2227, 2228, 2229, 2230, 2231, 2232, 2233, 2234, 2235, 2236, 2237, 2238, 2239, 2240, 2241, 2242, 2243, 2244, 2245, 2246, 2247, 2248, 2249, 2250, 2251, 2252, 2253, 2254, 2255, 2256, 2257, 2258, 2259, 2260, 2261, 2262, 2263, 2264, 2265, 2266, 2267, 2268, 2269, 2270, 2271, 2272, 2273, 2274, 2275, 2276, 2277, 2278, 2279, 2280, 2281, 2282, 2283, 2284, 2285, 2286, 2287, 2288, 2289, 2290, 2291, 2292, 2293, 2294, 2295, 2296, 2297, 2298, 2299, 2300, 2301, 2302, 2303, 2304, 2305, 2306, 2307, 2308, 2309, 2310, 2311, 2312, 2313, 2314, 2315, 2316, 2317, 2318, 2319, 2320, 2321, 2322, 2323, 2324, 2325, 2326, 2327, 2328, 2329, 2330, 2331, 2332, 2333, 2334, 2335, 2336, 2337, 2338, 2339, 2340, 2341, 2342, 2343, 2344, 2345, 2346, 2347, 2348, 2349, 2350, 2351, 2352, 2353, 2354, 2355, 2356, 2357, 2358, 2359, 2360, 2361, 2362, 2363, 2364, 2365, 2366, 2367, 2368, 2369, 2370, 2371, 2372, 2373, 2374, 2375, 2376, 2377, 2378, 2379, 2380, 2381, 2382, 2383, 2384, 2385, 2386, 2387, 2388, 2389, 2390, 2391, 2392, 2393, 2394, 2395, 2396, 2397, 2398, 2399, 2400, 2401, 2402, 2403, 2404, 2405, 2406, 2407, 2408, 2409, 2410, 2411, 2412, 2413, 2414, 2415, 2416, 2417, 2418, 2419, 2420, 2421, 2422, 2423, 2424, 2425, 2426, 2427, 2428, 2429, 2430, 2431, 2432, 2433, 2434, 2435, 2436, 2437, 2438, 2439, 2440, 2441, 2442, 2443, 2444, 2445, 2446, 2447, 2448, 2449, 2450, 2451, 2452, 2453, 2454, 2455, 2456, 2457, 2458, 2459, 2460, 2461, 2462, 2463, 2464, 2465, 2466, 2467, 2468, 2469, 2470, 2471, 2472, 2473, 2474, 2475, 2476, 2477, 2478, 2479, 2480, 2481, 2482, 2483, 2484, 2485, 2486, 2487, 2488, 2489, 2490, 2491, 2492, 2493, 2494, 2495, 2496, 2497, 2498, 2499, 2500, 2501, 2502, 2503, 2504, 2505, 2506, 2507, 2508, 2509, 2510, 2511, 2512, 2513, 2514, 2515, 2516, 2517, 2518, 2519, 2520, 2521, 2522, 2523, 2524, 2525, 2526, 2527, 2528, 2529, 2530, 2531, 2532, 2533, 2534, 2535, 2536, 2537, 2538, 2539, 2540, 2541, 2542, 2543, 2544, 2545, 2546, 2547, 2548, 2549, 2550, 2551, 2552, 2553, 2554, 2555, 2556, 2557, 2558, 2559, 2560, 2561, 2562, 2563, 2564, 2565, 2566, 2567, 2568, 2569, 2570, 2571, 2572, 2573, 2574, 2575, 2576, 2577, 2578, 2579, 2580, 2581, 2582, 2583, 2584, 2585, 2586, 2587, 2588, 2589, 2590, 2591, 2592, 2593, 2594, 2595, 2596, 2597, 2598, 2599, 2600, 2601, 2602, 2603, 2604, 2605, 2606, 2607, 2608, 2609, 2610, 2611, 2612, 2613, 2614, 2615, 2616, 2617, 2618, 2619, 2620, 2621, 2622, 2623, 2624, 2625, 2626, 2627, 2628, 2629, 2630, 2631, 2632, 2633, 2634, 2635, 2636, 2637, 2638, 2639, 2640, 2641, 2642, 2643, 2644, 2645, 2646, 2647, 2648, 2649, 2650, 2651, 2652, 2653, 2654, 2655, 2656, 2657, 2658, 2659, 2660, 2661, 2662, 2663, 2664, 2665, 2666, 2667, 2668, 2669, 2670, 2671, 2672, 2673, 2674, 2675, 2676, 2677, 2678, 2679, 26

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The particle shape shall be described as follows where length, width, and thickness refer to the greatest, intermediate, and least dimensions of a particle, respectively.

PARTICLE SHAPE

W=WIDTH
T=THICKNESS
L=LENGTH

A 3D perspective diagram of a rectangular particle. The top surface is labeled "PARTICLE". The dimensions are indicated by arrows: L for length, W for width, and H for height.

FLAT: $W/T > 3$
 ELONGATED: $L/W > 3$
 FLAT AND ELONGATED:
 -meets both criteria

FIG. 4 Criteria for Particle Shape

10.13 Additional comments shall be noted, such as the presence of roots or root holes, difficulty in drilling or augering

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TABLE 3 Criteria for Describing Moisture Condition

| Description | Criteria |
|-------------|---|
| Dry | Absence of moisture, dusty, dry to the touch |
| Moist | Damp but no visible water |
| Wet | Visible free water, usually soil is below water table |

TABLE 4 Criteria for Describing the Reaction With HCl

| Description | Criteria |
|-------------|--|
| None | No visible reaction |
| Weak | Some reaction, with bubbles forming slowly |
| Strong | Violent reaction, with bubbles forming immediately |

TABLE 5 Criteria for Describing Dilatancy

| Description | Criteria |
|-------------|--|
| Very soft | Thumb will penetrate soil more than 1 in. (25 mm) |
| Soft | Thumb will penetrate soil about 1 in. (25 mm) |
| Firm | Thumb will indent soil about 1/4 in. (6 mm) |
| Hard | Thumb will not indent soil but readily indented with thumbnail |
| Very hard | Thumbnail will not indent soil |

TABLE 6 Criteria for Describing Toughness

| Description | Criteria |
|-------------|---|
| Weak | Crumbles or breaks with handling or light finger pressure |
| Moderate | Crumbles or breaks with considerable finger pressure |
| Strong | Will not crumble or break with finger pressure |

TABLE 7 Criteria for Describing Dilatancy

| Description | Criteria |
|--------------|--|
| Stratified | Alternating layers of varying material or color with layers at least 6 mm thick; note thickness |
| Laminated | Alternating layers of varying material or color with the layers less than 6 mm thick; note thickness |
| Fissured | Breaks along definite planes of fracture with little resistance to fracturing |
| Slickensided | Fracture planes appear polished or glossy, sometimes striated |
| Blocky | Cohesive soil that can be broken down into small angular lumps which resist further breakdown |
| Lensed | Inclusion of small pockets of different soils, such as small lenses of sand scattered through a mass of clay; note thickness |
| Homogeneous | Same color and appearance throughout |

hole, caving of trench or hole, or the presence of mica.

10.14 A local or commercial name or a geologic interpretation of the soil, or both, may be added if identified as such.

10.15 A classification or identification of the soil in accordance with other classification systems may be added if identified as such.

11. Identification of Peat

11.1 A sample composed primarily of vegetable tissue in various stages of decomposition that has a fibrous to amorphous texture, usually a dark brown to black color, and an organic odor, shall be designated as a highly organic soil and shall be identified as peat, PT, and not subjected to the identification procedures described hereafter.

12. Preparation for Identification

12.1 The soil identification portion of this practice is based

on the portion of the soil sample that will pass a 3-in. (75-mm) sieve. The larger than 3-in. (75-mm) particles must be removed, manually, for a loose sample, or mentally, for an intact sample before classifying the soil.

12.2 Estimate and note the percentage of cobbles and the percentage of boulders. Performed visually, these estimates will be on the basis of volume percentage.

Note 9—Since the percentages of the particle-size distribution in Test Method D 2487 are by dry weight, and the estimates of percentages for gravel, sand, and fines in this practice are by dry weight, it is recommended that the report state that the percentages of cobbles and boulders are by volume.

12.3 Of the fraction of the soil smaller than 3 in. (75 mm), estimate and note the percentage, by dry weight, of the gravel, sand, and fines (see Appendix X4 for suggested procedures).

Note 10—Since the particle-size components appear visually on the basis of volume, considerable experience is required to estimate the percentages on the basis of dry weight. Frequent comparisons with laboratory particle-size analyses should be made.

12.3.1 The percentages shall be estimated to the closest 5 %. The percentages of gravel, sand, and fines must add up to 100 %.

12.3.2 If one of the components is present but not in sufficient quantity to be considered 5 % of the smaller than 3-in. (75-mm) portion, indicate its presence by the term *trace*, for example, trace of fines. A trace is not to be considered in the total of 100 % for the components.

13. Preliminary Identification

13.1 The soil is *fine grained* if it contains 50 % or more fines. Follow the procedures for identifying fine-grained soils of Section 14.

13.2 The soil is *coarse grained* if it contains less than 50 % fines. Follow the procedures for identifying coarse-grained soils of Section 15.

14. Procedure for Identifying Fine-Grained Soils

14.1 Select a representative sample of the material for examination. Remove particles larger than the No. 40 sieve (medium sand and larger) until a specimen equivalent to about a handful of material is available. Use this specimen for performing the dry strength, dilatancy, and toughness tests.

14.2 Dry Strength:

14.2.1 From the specimen, select enough material to mold into a ball about 1 in. (25 mm) in diameter. Mold the material until it has the consistency of putty, adding water if necessary.

14.2.2 From the molded material, make at least three test specimens. A test specimen shall be a ball of material about 1/2 in. (12 mm) in diameter. Allow the test specimens to dry in air, or sun, or by artificial means, as long as the temperature does not exceed 60°C.

14.2.3 If the test specimen contains natural dry lumps, those that are about 1/2 in. (12 mm) in diameter may be used in place of the molded balls.

Note 11—The process of molding and drying usually produces higher strengths than are found in natural dry lumps of soil.

14.2.4 Test the strength of the dry balls or lumps by crushing between the fingers. Note the strength as none, low,

medium, high, or very high in accordance with the criteria in Table 8. If natural dry lumps are used, do not use the results of any of the lumps that are found to contain particles of coarse sand.

14.2.5 The presence of high-strength water-soluble cementing materials, such as calcium carbonate, may cause exceptionally high dry strengths. The presence of calcium carbonate can usually be detected from the intensity of the reaction with dilute hydrochloric acid (see 10.6).

14.3 Dilatancy

14.3.1 From the specimen, select enough material to mold into a ball about 1/2 in. (12 mm) in diameter. Mold the material, adding water if necessary, until it has a soft, but not sticky, consistency.

14.3.2 Smooth the soil ball in the palm of one hand with the blade of a knife or small spatula. Shake horizontally, striking the side of the hand vigorously against the other hand several times. Note the reaction of water appearing on the surface of the soil. Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the criteria in Table 9. The reaction is the speed with which water appears while shaking, and disappears while squeezing.

14.4 Toughness

14.4.1 Following the completion of the dilatancy test, the test specimen is shaped into an elongated pat and rolled by hand on a smooth surface or between the palms into a thread about 1/8 in. (3 mm) in diameter. (If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation.) Fold the sample threads and re-roll repeatedly until the thread crumbles at a diameter of about 1/8 in. The thread will crumble at a diameter of 1/8 in. when the soil is near the plastic limit. Note the pressure required to roll the thread near the plastic limit. Also, note the strength of the thread. After the thread crumbles, the pieces should be lumped together and kneaded until the lump crumbles. Note the toughness of the material during kneading.

14.4.2 Describe the toughness of the thread and lump as low, medium, or high in accordance with the criteria in Table 10.

14.5 Plasticity—On the basis of observations made during the toughness test, describe the plasticity of the material in accordance with the criteria given in Table 11.

14.6 Decide whether the soil is an *inorganic* or an *organic* fine-grained soil (see 14.8). If inorganic, follow the steps given in 14.7.

TABLE 8 Criteria for Describing Toughness

| Description | Criteria |
|-------------|--|
| None | The dry specimen crumbles into powder with mere pressure of handling |
| Low | The dry specimen crumbles into powder with some finger pressure |
| Medium | The dry specimen breaks into pieces or crumbles with considerable finger pressure |
| High | The dry specimen cannot be broken with finger pressure. Specimen will break into pieces between thumb and a hard surface |
| Very high | The dry specimen cannot be broken between the thumb and a hard surface |

TABLE 9 Criteria for Describing Dilatancy

| Description | Criteria |
|-------------|---|
| None | No visible change in the specimen |
| Slow | Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing |
| Rapid | Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing |

TABLE 10 Criteria for Describing Toughness

| Description | Criteria |
|-------------|--|
| Low | Only slight pressure is required to roll the thread near the plastic limit. The thread and the lump are weak and soft |
| Medium | Medium pressure is required to roll the thread to near the plastic limit. The thread and the lump have medium stiffness |
| High | Considerable pressure is required to roll the thread to near the plastic limit. The thread and the lump have very high stiffness |

TABLE 11 Criteria for Describing Plasticity

| Description | Criteria |
|-------------|--|
| Nonplastic | A 1/8-in. (3-mm) thread cannot be rolled at any water content |
| Low | The thread can barely be rolled and the lump cannot be formed when drier than the plastic limit |
| Medium | The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be re-rolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit |
| High | It takes considerable time rolling and kneading to reach the plastic limit. The thread can be re-rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit |

14.7 Identification of Inorganic Fine-Grained Soils

14.7.1 Identify the soil as a *lean clay*, CL, if the soil has medium to high dry strength, no or slow dilatancy, and medium toughness and plasticity (see Table 12).

14.7.2 Identify the soil as a *fat clay*, CH, if the soil has high to very high dry strength, no dilatancy, and high toughness and plasticity (see Table 12).

14.7.3 Identify the soil as a *silt*, ML, if the soil has no to low dry strength, slow to rapid dilatancy, and low toughness and plasticity, or is nonplastic (see Table 12).

14.7.4 Identify the soil as an *elastic silt*, MH, if the soil has low to medium dry strength, no to slow dilatancy, and low to medium toughness and plasticity (see Table 12).

Note 12—These properties are similar to those for a lean clay. However, the silt will dry quickly on the hand and have a smooth, silky feel when dry. Some soils that would classify as MH in accordance with the criteria in Test Method D 2487 are visually difficult to distinguish from lean clays, CL. It may be necessary to perform laboratory testing for proper identification.

TABLE 12 Identification of Inorganic Fine-Grained Soils from Manual Tests

| Soil Symbol | Dry Strength | Dilatancy | Toughness |
|-------------|-------------------|---------------|--------------------------------|
| ML | None to low | Slow to rapid | Low or thread cannot be formed |
| CL | Medium to high | None to slow | Medium |
| MH | Low to medium | None to slow | Low to medium |
| CH | High to very high | None | High |

14.8 Identification of Organic Fine-Grained Soils:

14.8.1 Identify the soil as an *organic soil*, OL/OH, if the soil contains enough organic particles to influence the soil properties. Organic soils usually have a dark brown to black color and may have an organic odor. Often, organic soils will change color, for example, black to brown, when exposed to the air. Some organic soils will lighten in color significantly when air dried. Organic soils normally will not have a high toughness or plasticity. The thread for the toughness test will be spongy.

Note 13—In some cases, through practice and experience, it may be possible to further identify the organic soils as organic silts or organic clays, OL or OH. Correlations between the dilatancy, dry strength, toughness tests, and laboratory tests can be made to identify organic soils in certain deposits of similar materials of known geologic origin.

14.9 If the soil is estimated to have 15 to 25 % sand or gravel, or both, the words “with sand” or “with gravel” (whichever is more predominant) shall be added to the group name. For example: “lean clay with sand, CL” or “silt with gravel, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percentage of gravel, use “with sand.”

14.10 If the soil is estimated to have 30 % or more sand or gravel, or both, the words “sandy” or “gravelly” shall be added to the group name. Add the word “sandy” if there appears to be more sand than gravel. Add the word “gravelly” if there appears to be more gravel than sand. For example: “sandy lean clay, CL”, “gravelly fat clay, CH”, or “sandy silt, ML” (see Fig. 1a and Fig. 1b). If the percentage of sand is equal to the percent of gravel, use “sandy.”

15. Procedure for Identifying Coarse-Grained Soils

(Contains less than 50 % fines)

15.1 The soil is a *gravel* if the percentage of gravel is estimated to be more than the percentage of sand.

15.2 The soil is a *sand* if the percentage of gravel is estimated to be equal to or less than the percentage of sand.

15.3 The soil is a *clean gravel* or *clean sand* if the percentage of fines is estimated to be 5 % or less.

15.3.1 Identify the soil as a *well-graded gravel*, GW, or as a *well-graded sand*, SW, if it has a wide range of particle sizes and substantial amounts of the intermediate particle sizes.

15.3.2 Identify the soil as a *poorly graded gravel*, GP, or as a *poorly graded sand*, SP, if it consists predominantly of one size (uniformly graded), or it has a wide range of sizes with some intermediate sizes obviously missing (gap or skip graded).

15.4 The soil is either a *gravel with fines* or a *sand with fines* if the percentage of fines is estimated to be 15 % or more.

15.4.1 Identify the soil as a *clayey gravel*, GC, or a *clayey sand*, SC, if the fines are clayey as determined by the procedures in Section 14.

15.4.2 Identify the soil as a *silty gravel*, GM, or a *silty sand*, SM, if the fines are silty as determined by the procedures in Section 14.

15.5 If the soil is estimated to contain 10 % fines, give the soil a dual identification using two group symbols.

15.5.1 The first group symbol shall correspond to a clean gravel or sand (GW, GP, SW, SP) and the second symbol shall correspond to a gravel or sand with fines (GC, GM, SC, SM).

15.5.2 The group name shall correspond to the first group

symbol plus the words “with clay” or “with silt” to indicate the plasticity characteristics of the fines. For example: “well-graded gravel with clay, GW-GC” or “poorly graded sand with silt, SP-SM” (see Fig. 2).

15.6 If the specimen is predominantly sand or gravel but contains an estimated 15 % or more of the other coarse-grained constituent, the words “with gravel” or “with sand” shall be added to the group name. For example: “poorly graded gravel with sand, GP” or “clayey sand with gravel, SC” (see Fig. 2).

15.7 If the field sample contains any cobbles or boulders, or both, the words “with cobbles” or “with cobbles and boulders” shall be added to the group name. For example: “silty gravel with cobbles, GM.”

16. Report

16.1 The report shall include the information as to origin, and the items indicated in Table 13.

Note 14—Example: *Clayey Gravel with Sand and Cobbles, GC*—About 50 % fine to coarse, subrounded to subangular gravel; about 30 % fine to coarse, subrounded sand; about 20 % fines with medium plasticity, high dry strength, no dilatancy, medium toughness; weak reaction with HCl; original field sample had about 5 % (by volume) subrounded cobbles, maximum dimension, 150 mm

In-Place Conditions—Firm, homogeneous, dry, brown

Geologic Interpretation—Alluvial fan

Note 15—Other examples of soil descriptions and identification are given in Appendix X1 and Appendix X2.

Note 16—If desired, the percentages of gravel, sand, and fines may be stated in terms indicating a range of percentages, as follows:

Trace—Particles are present but estimated to be less than 5 %

Few—5 to 10 %

Little—15 to 25 %

Some—30 to 45 %

Abundant—50 to 100 %

TABLE 13 Checklist for Description of Soils

| | |
|---|--|
| 1. Group name | |
| 2. Group symbol | |
| 3. Percent of cobbles or boulders, or both (by volume) | |
| 4. Percent of gravel, sand, or fines, or all three (by dry weight) | |
| 5. Particle-size range: | Gravel—fine, coarse Sand—fine, medium, coarse |
| 6. Particle angularity: angular, subangular, subrounded, rounded | |
| 7. Particle shape: (if appropriate) flat, elongated, flat and elongated | |
| 8. Maximum particle size or dimension | |
| 9. Hardness of coarse sand and larger particles | |
| 10. Plasticity of fines: nonplastic, low, medium, high | |
| 11. Dry strength: none, low, medium, high, very high | |
| 12. Dilatancy: none, slow, rapid | |
| 13. Toughness: low, medium, high | |
| 14. Color (in moist condition) | |
| 15. Odor (mention only if organic or unusual) | |
| 16. Moisture: dry, moist, wet | |
| 17. Reaction with HCl: none, weak, strong | |
| For intact samples: | |
| 18. Consistency (fine-grained soils only): very soft, soft, firm, hard, very hard | |
| 19. Structure: stratified, laminated, fissured, slickensided, lensed, homogeneous | |
| 20. Cementation: weak, moderate, strong | |
| 21. Local name | |
| 22. Geologic interpretation | |
| 23. Additional comments: presence of roots or root holes; presence of mica, gypsum, etc.; surface coatings on coarse-grained particles; caving or sloughing of auger hole or trench sides; difficulty in augering or excavating; etc. | |

16.2 If, in the soil description, the soil is identified using a classification group symbol and name as described in Test Method D 2487, it must be distinctly and clearly stated in log forms, summary tables, reports, and the like, that the symbol and name are based on visual-manual procedures.

17. Precision and Bias

17.1 This practice provides qualitative information only.

therefore, a precision and bias statement is not applicable.

18. Keywords

18.1 classification; clay; gravel; organic soils; sand; silt; soil classification; soil description; visual classification

APPENDIXES

(Nonmandatory Information)

X1. EXAMPLES OF VISUAL SOIL DESCRIPTIONS

X1.1 The following examples show how the information required in 16.1 can be reported. The information that is included in descriptions should be based on individual circumstances and need.

X1.1.1 *Well-Graded Gravel with Sand (GW)*—About 75 % fine to coarse, hard, subangular gravel; about 25 % fine to coarse, hard, subangular sand; trace of fines; maximum size, 75 mm, brown, dry; no reaction with HCl.

X1.1.2 *Silty Sand with Gravel (SM)*—About 60 % predominantly fine sand; about 25 % silty fines with low plasticity, low dry strength, rapid dilatancy, and low toughness; about 15 % fine, hard, subrounded gravel, a few gravel-size particles fractured with hammer blow; maximum size, 25 mm; no reaction with HCl (Note—Field sample size smaller than recommended).

In-Place Conditions—Firm, stratified and contains lenses of silt 1 to 2 in. (25 to 50 mm) thick, moist, brown to gray; in-place density 106 lb/ft³; in-place moisture 9 %.

X1.1.3 *Organic Soil (OL/OH)*—About 100 % fines with low plasticity, slow dilatancy, low dry strength, and low toughness; wet, dark brown, organic odor; weak reaction with HCl.

X1.1.4 *Silty Sand with Organic Fines (SM)*—About 75 % fine to coarse, hard, subangular reddish sand; about 25 % organic and silty dark brown nonplastic fines with no dry strength and slow dilatancy; wet; maximum size, coarse sand; weak reaction with HCl.

X1.1.5 *Poorly Graded Gravel with Silt, Sand, Cobbles and Boulders (GP-GM)*—About 75 % fine to coarse, hard, subrounded to subangular gravel; about 15 % fine, hard, subrounded to subangular sand; about 10 % silty nonplastic fines; moist, brown; no reaction with HCl; original field sample had about 5 % (by volume) hard, subrounded cobbles and a trace of hard, subrounded boulders, with a maximum dimension of 18 in. (450 mm).

X2. USING THE IDENTIFICATION PROCEDURE AS A DESCRIPTIVE SYSTEM FOR SHALE, CLAYSTONE, SHELLS, SLAG, CRUSHED ROCK, AND THE LIKE

X2.1 The identification procedure may be used as a descriptive system applied to materials that exist in-situ as shale, claystone, sandstone, siltstone, mudstone, etc., but convert to soils after field or laboratory processing (crushing, slaking, and the like).

X2.2 Materials such as shells, crushed rock, slag, and the like, should be identified as such. However, the procedures used in this practice for describing the particle size and plasticity characteristics may be used in the description of the material. If desired, an identification using a group name and symbol according to this practice may be assigned to aid in describing the material.

X2.3 The group symbol(s) and group names should be placed in quotation marks or noted with some type of distinguishing symbol. See examples.

X2.4 Examples of how group names and symbols can be incorporated into a descriptive system for materials that are not

naturally occurring soils are as follows:

X2.4.1 *Shale Chunks*—Retrieved as 2 to 4-in. (50 to 100-mm) pieces of shale from power auger hole, dry, brown, no reaction with HCl. After slaking in water for 24 h, material identified as "Sandy Lean Clay (CL)"; about 60 % fines with medium plasticity, high dry strength, no dilatancy, and medium toughness; about 35 % fine to medium, hard sand; about 5 % gravel-size pieces of shale.

X2.4.2 *Crushed Sandstone*—Product of commercial crushing operation: "Poorly Graded Sand with Silt (SP-SM)"; about 90 % fine to medium sand; about 10 % nonplastic fines; dry, reddish-brown, strong reaction with HCl.

X2.4.3 *Broken Shells*—About 60 % gravel-size broken shells; about 30 % sand and sand-size shell pieces; about 10 % fines; "Poorly Graded Gravel with Sand (GP)."

X2.4.4 *Crushed Rock*—Processed from gravel and cobbles in Pit No. 7; "Poorly Graded Gravel (GP)"; about 90 % fine, hard, angular gravel-size particles; about 10 % coarse, hard,

angular sand-size particles; dry, tan; no reaction with HCl.

X3. SUGGESTED PROCEDURE FOR USING A BORDERLINE SYMBOL FOR SOILS WITH TWO POSSIBLE IDENTIFICATIONS.

X3.1 Since this practice is based on estimates of particle size distribution and plasticity characteristics, it may be difficult to clearly identify the soil as belonging to one category. To indicate that the soil may fall into one of two possible basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example: SC/CL or CL/CH.

X3.1.1 A borderline symbol may be used when the percentage of fines is estimated to be between 45 and 55 %. One symbol should be for a coarse-grained soil with fines and the other for a fine-grained soil. For example: GM/ML or CL/SC.

X3.1.2 A borderline symbol may be used when the percentage of sand and the percentage of gravel are estimated to be about the same. For example: GP/SP, SC/GC, GM/SM. It is practically impossible to have a soil that would have a borderline symbol of GW/SW.

X3.1.3 A borderline symbol may be used when the soil could be either well graded or poorly graded. For example: GW/GP, SW/SP.

X3.1.4 A borderline symbol may be used when the soil could either be a silt or a clay. For example: CL/ML, CH/MH, SC/SM.

X3.1.5 A borderline symbol may be used when a fine-grained soil has properties that indicate that it is at the boundary between a soil of low compressibility and a soil of high compressibility. For example: CL/CH, MH/ML.

X3.2 The order of the borderline symbols should reflect similarity to surrounding or adjacent soils. For example: soils in a borrow area have been identified as CH. One sample is considered to have a borderline symbol of CL and CH. To show similarity, the borderline symbol should be CH/CL.

X3.3 The group name for a soil with a borderline symbol should be the group name for the first symbol, except for:

CL/CH lean to fat clay

ML/CL clayey silt

CL/ML silty clay

X3.4 The use of a borderline symbol should not be used indiscriminately. Every effort shall be made to first place the soil into a single group.

X4. SUGGESTED PROCEDURES FOR ESTIMATING THE PERCENTAGES OF GRAVEL, SAND, AND FINES IN A SOIL SAMPLE

X4.1 *Jar Method*—The relative percentage of coarse- and fine-grained material may be estimated by thoroughly shaking a mixture of soil and water in a test tube or jar, and then allowing the mixture to settle. The coarse particles will fall to the bottom and successively finer particles will be deposited with increasing time; the sand sizes will fall out of suspension in 20 to 30 s. The relative proportions can be estimated from the relative volume of each size separate. This method should be correlated to particle-size laboratory determinations.

X4.2 *Visual Method*—Mentally visualize the gravel size particles placed in a sack (or other container) or sacks. Then, do the same with the sand size particles and the fines. Then, mentally compare the number of sacks to estimate the percentage of this No. 4 sieve size and minus No. 4 sieve size present.

The percentages of sand and fines in the minus sieve size No. 4 material can then be estimated from the wash test (X4.3).

X4.3 *Wash Test (for relative percentages of sand and fines)*—Select and moisten enough minus No. 4 sieve size material to form a 1-in (25-mm) cube of soil. Cut the cube in half, set one-half to the side, and place the other half in a small dish. Wash and decant the fines out of the material in the dish until the wash water is clear and then compare the two samples and estimate the percentage of sand and fines. Remember that the percentage is based on weight, not volume. However, the volume comparison will provide a reasonable indication of grain size percentages.

X4.3.1 While washing, it may be necessary to break down lumps of fines with the finger to get the correct percentages.

X5. ABBREVIATED SOIL CLASSIFICATION SYMBOLS

X5.1 In some cases, because of lack of space, an abbreviated system may be useful to indicate the soil classification symbol and name. Examples of such cases would be graphical logs, databases, tables, etc.

X5.2 This abbreviated system is not a substitute for the full name and descriptive information but can be used in supplementary presentations when the complete description is referenced.

X5.3 The abbreviated system should consist of the soil classification symbol based on this standard with appropriate lower case letter prefixes and suffixes as:

Prefix:

Suffix:

| | | Group Symbol and Full Name | Abbreviated |
|--------------|-------------------|---|-------------|
| s = sandy | s = with sand | CL, Sandy lean clay | s(CL) |
| g = gravelly | g = with gravel | SP-SM, Poorly graded sand with silt and gravel | (SP-SM)g |
| | c = with cobbles | GP, poorly graded gravel with sand, cobbles, and boulders | (GP)scb |
| | b = with boulders | ML, gravelly silt with sand and cobbles | g(ML)sc |

X5.4 The soil classification symbol is to be enclosed in parenthesis. Some examples would be:

SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (1993⁽¹⁾) that may impact the use of this standard.

(1) Added Practice D 3740 to Section 2.

(2) Added Note 5 under 5.7 and renumbered subsequent notes.

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Designation: D 1586 – 08

Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils¹

This standard is issued under the fixed designation D 1586; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 This test method describes the procedure, generally known as the Standard Penetration Test (SPT), for driving a split-barrel sampler to obtain a representative disturbed soil sample for identification purposes, and measure the resistance of the soil to penetration of the sampler. Another method (Test Method D 3550) to drive a split-barrel sampler to obtain a representative soil sample is available but the hammer energy is not standardized.

1.2 Practice D 6026 gives a guide to determining the normalized penetration resistance of sands for energy adjustments of N-value to a constant energy level for evaluating liquefaction potential.

1.3 Test results and identification information are used to estimate subsurface conditions for foundation design.

1.4 Penetration resistance testing is typically performed at 5-foot depth intervals or when a significant change of materials is observed during drilling, unless otherwise specified.

1.5 This test method is limited to use in nonlithified soils and soils whose maximum particle size is approximately less than one-half of the sampler diameter.

1.6 This test method involves use of rotary drilling equipment (Guide D 5783, Practice D 6151). Other drilling and sampling procedures (Guide D 6286, Guide D 6169) are available and may be more appropriate. Considerations for hand driving or shallow sampling without boreholes are not addressed. Subsurface investigations should be recorded in accordance with Practice D 5434. Samples should be preserved and transported in accordance with Practice D 4220 using Group B. Soil samples should be identified by group name and symbol in accordance with Practice D 2488.

1.7 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D 6026, unless superseded by this test method.

1.8 The values stated in inch-pound units are to be regarded as standard, except as noted below. The values given in

parentheses are mathematical conversions to SI units, which are provided for information only and are not considered standard.

1.8.1 The gravitational system of inch-pound units is used when dealing with inch-pound units. In this system, the pound (lbf) represents a unit of force (weight), while the unit for mass is slugs.

1.9 Penetration resistance measurements often will involve safety planning, administration, and documentation. This test method does not purport to address all aspects of exploration and site safety. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Performance of the test usually involves use of a drill rig; therefore, safety requirements as outlined in applicable safety standards (for example, OSHA regulations,² NDA Drilling Safety Guide,³ drilling safety manuals, and other applicable state and local regulations) must be observed.

2. Referenced Documents

2.1 ASTM Standards:⁴

D 653 Terminology Relating to Soil, Rock, and Contained Fluids

D 854 Test Methods for Specific Gravity of Soil Solids by Water Pycnometer

D 1587 Practice for Thin-Walled Tube Sampling of Soils for Geotechnical Purposes

D 2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

D 2487 Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System)

D 2488 Practice for Description and Identification of Soils

² Available from Occupational Safety and Health Administration (OSHA), 200 Constitution Ave., NW, Washington, DC 20210, <http://www.osha.gov>

³ Available from the National Drilling Association, 3511 Center Rd., Suite 8, Brunswick, OH 44212, <http://www.ndafo.com>.

⁴ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

¹ This method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.02 on Sampling and Related Field Testing for Soil Evaluations.

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*A Summary of Changes section appears at the end of this standard.

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(Visual-Manual Procedure)

- D 3550 Practice for Thick Wall, Ring-Lined, Split Barrel, Drive Sampling of Soils
- D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
- D 4220 Practices for Preserving and Transporting Soil Samples
- D 4633 Test Method for Energy Measurement for Dynamic Penetrometers
- D 5434 Guide for Field Logging of Subsurface Explorations of Soil and Rock
- D 5783 Guide for Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices
- D 6026 Practice for Using Significant Digits in Geotechnical Data
- D 6066 Practice for Determining the Normalized Penetration Resistance of Sands for Evaluation of Liquefaction Potential
- D 6151 Practice for Using Hollow-Stem Augers for Geotechnical Exploration and Soil Sampling
- D 6169 Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations
- D 6286 Guide for Selection of Drilling Methods for Environmental Site Characterization
- D 6913 Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis

3. Terminology

3.1 *Definitions:* Definitions of terms included in Terminology D 683 specific to this practice are:

3.1.1 *cathead, n*—the rotating drum or windlass in the rope-cathead lift system around which the operator wraps a rope to lift and drop the hammer by successively tightening and loosening the rope turns around the drum.

3.1.2 *drill rods, n*—rods used to transmit downward force and torque to the drill bit while drilling a borehole.

3.1.3 *N-value, n*—the blow count representation of the penetration resistance of the soil. The *N-value*, reported in blows per foot, equals the sum of the number of blows (*N*) required to drive the sampler over the depth interval of 6 to 18 in. (150 to 450 mm) (see 7.3).

3.1.4 *Standard Penetration Test (SPT), n*—a test process in the bottom of the borehole where a split-barrel sampler having an inside diameter of either 1-1/2-in. (38.1 mm) or 1-3/8-in. (34.9 mm) (see Note 2) is driven a given distance of 1.0 ft (0.30 m) after a seating interval of 0.5 ft (0.15 m) using a hammer weighing approximately 140-lbf (623-N) falling 30 ± 1.0 in. (0.76 m \pm 0.030 m) for each hammer blow.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *anvil, n*—that portion of the drive-weight assembly which the hammer strikes and through which the hammer energy passes into the drill rods.

3.2.2 *drive weight assembly, n*—an assembly that consists of the hammer, anvil, hammer fall guide system, drill rod attachment system, and any hammer drop system hoisting attachments.

3.2.3 *hammer, n*—that portion of the drive-weight assembly consisting of the 140 ± 2 lbf (623 \pm 9 N) impact weight which is successively lifted and dropped to provide the energy that accomplishes the sampling and penetration.

3.2.4 *hammer drop system, n*—that portion of the drive-weight assembly by which the operator or automatic system accomplishes the lifting and dropping of the hammer to produce the blow.

3.2.5 *hammer fall guide, n*—that part of the drive-weight assembly used to guide the fall of the hammer.

3.2.6 *number of rope turns, n*—the total contact angle between the rope and the cathead at the beginning of the operator's rope slackening to drop the hammer, divided by 360° (see Fig. 1).

3.2.7 *sampling rods, n*—rods that connect the drive-weight assembly to the sampler. Drill rods are often used for this purpose.

4. Significance and Use

4.1 This test method provides a disturbed soil sample for moisture content determination, for identification and classification (Practices D 2487 and D 2488) purposes, and for laboratory tests appropriate for soil obtained from a sampler that will produce large shear strain disturbance in the sample such as Test Methods D 854, D 2216, and D 6913. Soil deposits containing gravels, cobbles, or boulders typically result in penetration refusal and damage to the equipment.

4.2 This test method provides a disturbed soil sample for moisture content determination and laboratory identification. Sample quality is generally not suitable for advanced laboratory testing for engineering properties. The process of driving the sampler will cause disturbance of the soil and change the engineering properties. Use of the thin wall tube sampler (Practice D 1587) may result in less disturbance in soft soils. Coring techniques may result in less disturbance than SPT sampling for harder soils, but it is not always the case, that is, some cemented soils may become loosened by water action during coring; see Practice D 6151, and Guide D 6169.

4.3 This test method is used extensively in a great variety of geotechnical exploration projects. Many local correlations and widely published correlations which relate blow count, or *N-value*, and the engineering behavior of earthworks and foundations are available. For evaluating the liquefaction potential of sands during an earthquake event, the *N-value* should be normalized to a standard overburden stress level. Practice D 6066 provides methods to obtain a record of normalized resistance of sands to the penetration of a standard sampler driven by a standard energy. The penetration resistance is adjusted to drill rod energy ratio of 60 % by using a hammer system with either an estimated energy delivery or directly measuring drill rod stress wave energy using Test Method D 4633.

Note 1—The reliability of data and interpretations generated by this practice is dependent on the competence of the personnel performing it

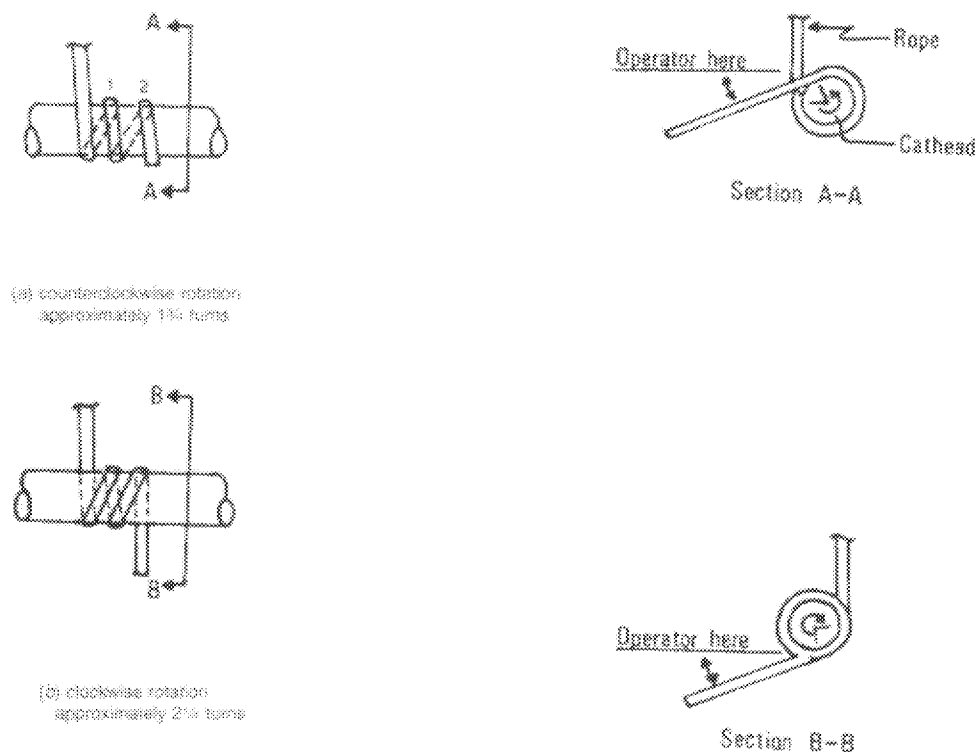


FIG. 1 Definitions of the Number of Rope Turns and the Angle for (a) Counterclockwise Rotation and (b) Clockwise Rotation of the Cathead

and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D 3740 generally are considered capable of competent testing. Users of this practice are cautioned that compliance with Practice D 3740 does not assure reliable testing. Reliable testing depends on several factors and Practice D 3740 provides a means of evaluating some of these factors. Practice D 3740 was developed for agencies engaged in the testing, inspection, or both, of soils and rock. As such, it is not totally applicable to agencies performing this practice. Users of this test method should recognize that the framework of Practice D 3740 is appropriate for evaluating the quality of an agency performing this test method. Currently, there is no known qualifying national authority that inspects agencies that perform this test method.

5. Apparatus

5.1 Drilling Equipment—Any drilling equipment that provides at the time of sampling a suitable borehole before insertion of the sampler and ensures that the penetration test is performed on undisturbed soil shall be acceptable. The following pieces of equipment have proven to be suitable for advancing a borehole in some subsurface conditions:

5.1.1 Drag, Chopping, and Fishtail Bits, less than 6½ in. (165 mm) and greater than 2¼ in. (57 mm) in diameter may be used in conjunction with open-hole rotary drilling or casing-advancement drilling methods. To avoid disturbance of the underlying soil, bottom discharge bits are not permitted; only side discharge bits are permitted.

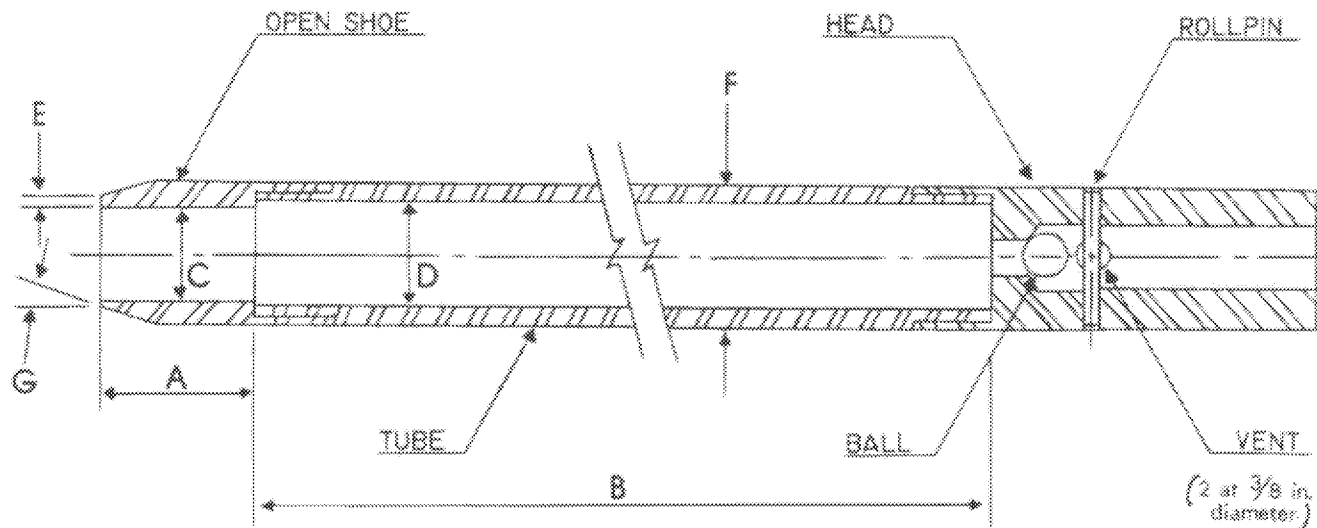
5.1.2 Roller-Cone Bits, less than 6½ in. (165 mm) and greater than 2¼ in. (57 mm) in diameter may be used in conjunction with open-hole rotary drilling or casing-advancement drilling methods if the drilling fluid discharge is deflected.

5.1.3 Hollow-Stem Continuous Flight Augers, with or without a center bit assembly, may be used to drill the borehole. The inside diameter of the hollow-stem augers shall be less than 6½ in. (165 mm) and not less than 2¼ in. (57 mm).

5.1.4 Solid, Continuous Flight, Bucket and Hand Augers, less than 6½ in. (165 mm) and not less than 2¼ in. (57 mm) in diameter may be used if the soil on the side of the borehole does not cave onto the sampler or sampling rods during sampling.

5.2 Sampling Rods—Flush-joint steel drill rods shall be used to connect the split-barrel sampler to the drive-weight assembly. The sampling rod shall have a stiffness (moment of inertia) equal to or greater than that of parallel wall “A” rod (a steel rod that has an outside diameter of 1-5/8 in. (41.3 mm) and an inside diameter of 1-1/8 in. (28.5 mm)).

5.3 Split-Barrel Sampler—The standard sampler dimensions are shown in Fig. 2. The sampler has an outside diameter of 2.00 in. (50.8 mm). The inside diameter of the of the split-barrel (dimension D in Fig. 2) can be either 1½-in. (38.1



A = 1.0 to 2.0 in. (25 to 50 mm)
 B = 18.0 to 30.0 in. (0.457 to 0.762 m)
 C = 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
 D = 1.60 ± 0.05 – 0.00 in. (38.1 ± 1.3 – 0.0 mm)
 E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
 F = 2.00 ± 0.05 – 0.00 in. (50.8 ± 1.3 – 0.0 mm)
 G = 16.0° to 22.0°

FIG. 2 Split-Barrel Sampler

mm) or 1½-in. (34.9 mm) (see Note 2). A 16-gauge liner can be used inside the 1½-in. (38.1 mm) split barrel sampler. The driving shoe shall be of hardened steel and shall be replaced or repaired when it becomes dented or distorted. The penetrating end of the drive shoe may be slightly rounded. The split-barrel sampler must be equipped with a ball check and vent. Metal or plastic baskets may be used to retain soil samples.

NOTE 2—Both theory and available test data suggest that *N*-values may differ as much as 10 to 30 % between a constant inside diameter sampler and upset wall sampler. If it is necessary to correct for the upset wall sampler refer to Practice D1086. In North America, it is now common practice to use an upset wall sampler with an inside diameter of 1½ in. At one time, liners were used but practice evolved to use the upset wall sampler without liners. Use of an upset wall sampler allows for use of retainers if needed, reduces inside friction, and improves recovery. Many other countries still use a constant ID split-barrel sampler, which was the original standard and still acceptable within this standard.

5.4 Drive-Weight Assembly

5.4.1 Hammer and Anvil—The hammer shall weigh 140 ± 2 lbf (623 ± 9 N) and shall be a rigid metallic mass. The hammer shall strike the anvil and make steel on steel contact when it is dropped. A hammer fall guide permitting an unimpeded fall shall be used. Fig. 3 shows a schematic of such hammers. Hammers used with the cathead and rope method shall have an unimpeded over lift capacity of at least 4 in. (100 mm). For safety reasons, the use of a hammer assembly with an internal anvil is encouraged as shown in Fig. 3. The total mass of the hammer assembly bearing on the drill rods should not be more than 250 ± 10 lbf (113 ± 5 kg).

NOTE 3—It is suggested that the hammer fall guide be permanently marked to enable the operator or inspector to judge the hammer drop height.

5.4.2 Hammer Drop System—Rope-cathead, trip, semi-automatic or automatic hammer drop systems, as shown in Fig. 4 may be used, providing the lifting apparatus will not cause penetration of the sampler while re-engaging and lifting the hammer.

5.5 Accessory Equipment—Accessories such as labels, sample containers, data sheets, and groundwater level measuring devices shall be provided in accordance with the requirements of the project and other ASTM standards.

6. Drilling Procedure

6.1 The borehole shall be advanced incrementally to permit intermittent or continuous sampling. Test intervals and locations are normally stipulated by the project engineer or geologist. Typically, the intervals selected are 5 ft (1.5 m) or less in homogeneous strata with test and sampling locations at every change of strata. Record the depth of drilling to the nearest 0.1 ft (0.030 m).

6.2 Any drilling procedure that provides a suitably clean and stable borehole before insertion of the sampler and assures that the penetration test is performed on essentially undisturbed soil shall be acceptable. Each of the following procedures has proven to be acceptable for some subsurface conditions. The subsurface conditions anticipated should be considered when selecting the drilling method to be used.

6.2.1 Open-hole rotary drilling method.

6.2.2 Continuous flight hollow-stem auger method.

6.2.3 Wash boring method.

6.2.4 Continuous flight solid auger method.

6.3 Several drilling methods produce unacceptable boreholes. The process of jetting through an open tube sampler and

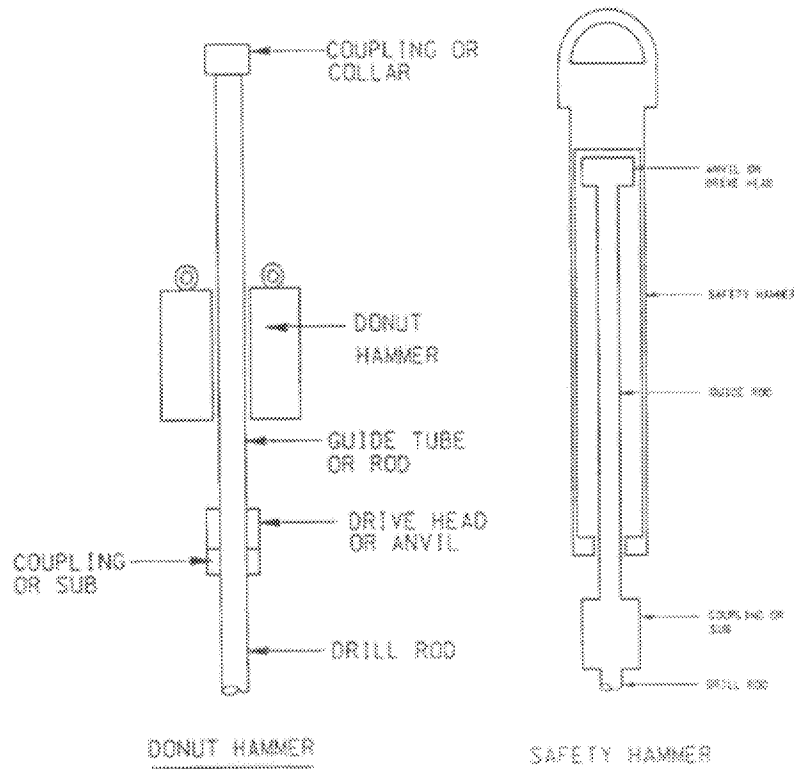


FIG. 3 Schematic Drawing of the Donut Hammer and Safety Hammer

then sampling when the desired depth is reached shall not be permitted. The continuous flight solid auger method shall not be used for advancing the borehole below a water table or below the upper confining bed of a confined non-cohesive stratum that is under artesian pressure. Casing may not be advanced below the sampling elevation prior to sampling. Advancing a borehole with bottom discharge bits is not permissible. It is not permissible to advance the borehole for subsequent insertion of the sampler solely by means of previous sampling with the SPT sampler.

6.4 The drilling fluid level within the borehole or hollow-stem augers shall be maintained at or above the in situ groundwater level at all times during drilling, removal of drill rods, and sampling.

7. Sampling and Testing Procedure

7.1 After the borehole has been advanced to the desired sampling elevation and excessive cuttings have been removed, record the cleanout depth to the nearest 0.1 ft (0.030 m), and prepare for the test with the following sequence of operations:

7.1.1 Attach either split-barrel sampler Type A or B to the sampling rods and lower into the borehole. Do not allow the sampler to drop onto the soil to be sampled.

7.1.2 Position the hammer above and attach the anvil to the top of the sampling rods. This may be done before the sampling rods and sampler are lowered into the borehole.

7.1.3 Rest the dead weight of the sampler, rods, anvil, and drive weight on the bottom of the borehole. Record the sampling start depth to the nearest 0.1 ft (0.030 m). Compare

the sampling start depth to the cleanout depth in 7.1. If excessive cuttings are encountered at the bottom of the borehole, remove the sampler and sampling rods from the borehole and remove the cuttings.

7.1.4 Mark the drill rods in three successive 0.5-foot (0.15 m) increments so that the advance of the sampler under the impact of the hammer can be easily observed for each 0.5-foot (0.15 m) increment.

7.2 Drive the sampler with blows from the 140-lbf (623-N) hammer and count the number of blows applied in each 0.5-foot (0.15-m) increment until one of the following occurs:

7.2.1 A total of 50 blows have been applied during any one of the three 0.5-foot (0.15-m) increments described in 7.1.4.

7.2.2 A total of 100 blows have been applied.

7.2.3 There is no observed advance of the sampler during the application of 10 successive blows of the hammer.

7.2.4 The sampler is advanced the complete 1.5 ft (0.45 m) without the limiting blow counts occurring as described in 7.2.1, 7.2.2, or 7.2.3.

7.2.5 If the sampler sinks under the weight of the hammer, weight of rods, or both, record the length of travel to the nearest 0.1 ft (0.030 m), and drive the sampler through the remainder of the test interval. If the sampler sinks the complete interval, stop the penetration, remove the sampler and sampling rods from the borehole, and advance the borehole through the very soft or very loose materials to the next desired sampling elevation. Record the *N*-value as either weight of hammer, weight of rods, or both.

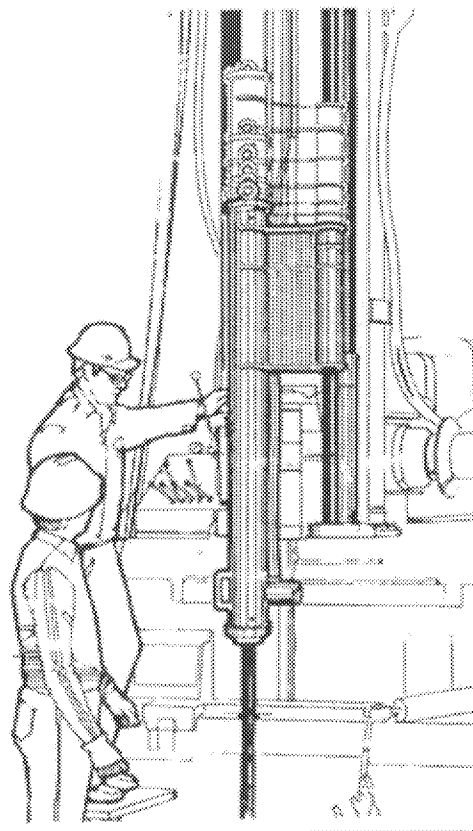


FIG. 4 Automatic Trip Hammer

7.3 Record the number of blows (N) required to advance the sampler each 0.5-foot (0.15 m) of penetration or fraction thereof. The first 0.5-foot (0.15 m) is considered to be a seating drive. The sum of the number of blows required for the second and third 0.5-foot (0.15 m) of penetration is termed the "standard penetration resistance," or the " N -value." If the sampler is driven less than 1.5 ft (0.45 m), as permitted in 7.2.1, 7.2.2, or 7.2.3, the number of blows per each complete 0.5-foot (0.15 m) increment and per each partial increment shall be recorded on the boring log. For partial increments, the depth of penetration shall be reported to the nearest 0.1 ft (0.030 m) in addition to the number of blows. If the sampler advances below the bottom of the borehole under the static weight of the drill rods or the weight of the drill rods plus the static weight of the hammer, this information should be noted on the boring log.

7.4 The raising and dropping of the 140-lbf (623-N) hammer shall be accomplished using either of the following two methods. Energy delivered to the drill rod by either method can be measured according to procedures in Test Method D 4633.

7.4.1 *Method A*—By using a trip, automatic, or semi-automatic hammer drop system that lifts the 140-lbf (623-N) hammer and allows it to drop 30 ± 1.0 in. (0.76 m \pm 0.030 m) with limited unimpedance. Drop heights adjustments for automatic and trip hammers should be checked daily and at first indication of variations in performance. Operation of automatic hammers shall be in strict accordance with operations manuals.

7.4.2 *Method B*—By using a cathead to pull a rope attached to the hammer. When the cathead and rope method is used the system and operation shall conform to the following:

7.4.2.1 The cathead shall be essentially free of rust, oil, or grease and have a diameter in the range of 6 to 10 in. (150 to 250 mm).

7.4.2.2 The cathead should be operated at a minimum speed of rotation of 100 RPM.

7.4.2.3 The operator should generally use either 1-3/4 or 2-1/4 rope turns on the cathead, depending upon whether or not the rope comes off the top (1-3/4 turns for counterclockwise rotation) or the bottom (2-1/4 turns for clockwise rotation) of the cathead during the performance of the penetration test, as shown in Fig. 1. It is generally known and accepted that 2-3/4 or more rope turns considerably impedes the fall of the hammer and should not be used to perform the test. The cathead rope should be stiff, relatively dry, clean, and should be replaced when it becomes excessively frayed, oily, limp, or burned.

7.4.2.4 For each hammer blow, a 30 ± 1.0 in. (0.76 m \pm 0.030 m) lift and drop shall be employed by the operator. The operation of pulling and throwing the rope shall be performed rhythmically without holding the rope at the top of the stroke.

NOTE 4—If the hammer drop height is something other than 30 ± 1.0 in. (0.76 m \pm 0.030 m), then record the new drop height. For soils other than sands, there is no known data or research that relates to adjusting the N -value obtained from different drop heights. Test method D 4633 provides information on making energy measurement for variable drop

heights and Practice D 6966 provides information on adjustment of N -value to a constant energy level (60 % of theoretical, N_{60}). Practice D 6966 allows the hammer drop height to be adjusted to provide 60 % energy.

7.5 Bring the sampler to the surface and open. Record the percent recovery to the nearest 1 % or the length of sample recovered to the nearest 0.01 ft (5 mm). Classify the soil samples recovered as to, in accordance with Practice D 2488, then place one or more representative portions of the sample into sealable moisture-proof containers (jars) without ramming or distorting any apparent stratification. Seal each container to prevent evaporation of soil moisture. Affix labels to the containers bearing job designation, boring number, sample depth, and the blow count per 0.5-foot (0.15-m) increment. Protect the samples against extreme temperature changes. If there is a soil change within the sampler, make a jar for each stratum and note its location in the sampler barrel. Samples should be preserved and transported in accordance with Practice D 4220 using Group B.

8. Data Sheet(s)/Form(s)

8.1 Data obtained in each borehole shall be recorded in accordance with the Subsurface Logging Guide D 5434 as required by the exploration program. An example of a sample data sheet is included in Appendix X1.

8.2 Drilling information shall be recorded in the field and shall include the following:

- 8.2.1 Name and location of job.
- 8.2.2 Names of crew.
- 8.2.3 Type and make of drilling machine.
- 8.2.4 Weather conditions.
- 8.2.5 Date and time of start and finish of borehole.
- 8.2.6 Boring number and location (station and coordinates, if available and applicable).
- 8.2.7 Surface elevation, if available.
- 8.2.8 Method of advancing and cleaning the borehole.
- 8.2.9 Method of keeping borehole open.
- 8.2.10 Depth of water surface to the nearest 0.1 ft (0.030 m) and drilling depth to the nearest 0.1 ft (0.030 m) at the time of a noted loss of drilling fluid, and time and date when reading or notation was made.
- 8.2.11 Location of strata changes, to the nearest 0.5 ft (15 cm).
- 8.2.12 Size of casing, depth of cased portion of borehole to the nearest 0.1 ft (0.030 m).

- 8.2.13 Equipment and Method A or B of driving sampler.
- 8.2.14 Sampler length and inside diameter of barrel, and if a sample basket retainer is used.
- 8.2.15 Size, type, and section length of the sampling rods, and
- 8.2.16 Remarks.

8.3 Data obtained for each sample shall be recorded in the field and shall include the following:

- 8.3.1 Top of sample depth to the nearest 0.1 ft (0.030 m) and, if utilized, the sample number.
- 8.3.2 Description of soil.
- 8.3.3 Strata changes within sample.
- 8.3.4 Sampler penetration and recovery lengths to the nearest 0.1 ft (0.030 m), and
- 8.3.5 Number of blows per 0.5 foot (0.15 m) or partial increment.

9. Precision and Bias

9.1 *Precision*—Test data on precision is not presented due to the nature of this test method. It is either not feasible or too costly at this time to have ten or more agencies participate in an in situ testing program at a given site.

9.1.1 The Subcommittee 18.02 is seeking additional data from the users of this test method that might be used to make a limited statement on precision. Present knowledge indicates the following:

9.1.1.1 Variations in N -values of 100 % or more have been observed when using different standard penetration test apparatus and drillers for adjacent boreholes in the same soil formation. Current opinion, based on field experience, indicates that when using the same apparatus and driller, N -values in the same soil can be reproduced with a coefficient of variation of about 10 %.

9.1.1.2 The use of faulty equipment, such as an extremely massive or damaged anvil, a rusty cathead, a low speed cathead, an old, oily rope, or massive or poorly lubricated rope sheaves can significantly contribute to differences in N -values obtained between operator-drill rig systems.

9.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.

10. Keywords

10.1 blow count; in-situ test; penetration resistance; soil; split-barrel sampling; standard penetration test

APPENDIX

(Nonmandatory Information)

XI. Example Data Sheet

X1.1 See Fig. 5.

FIG. 5 Example Data Sheet



SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this standard since the last issue (D 1586 – 99) that may impact the use of this standard. (Approved February 1, 2008.)

- | | |
|--|--|
| <p>(1) There have been numerous changes to this standard to list them separately. From the most recent main ballot process, additional changes were requested and incorporated into this newest revision. Stated below is a highlight of some of the changes.</p> <p>(2) Scope was completely revised.</p> <p>(3) Referenced Documents updated to include new standards.</p> | <p>(4) Terminology: added section on Definitions.</p> <p>(5) Significance and Use: clarified use of the SPT test.</p> <p>(6) Apparatus: general editorial changes.</p> <p>(7) Sampling and Testing Procedure: general editorial changes.</p> <p>(8) Data Sheets/Forms: general editorial changes.</p> <p>(9) Precision and Bias: added Sections 9.1.1.1 and 9.1.1.2.</p> |
|--|--|

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Table 1
EXAMPLE SOIL DESCRIPTIONS

POORLY GRADED SAND (SP), light brown, moist, loose, fine sand size

FAT CLAY (CH), dark gray, moist, stiff

SILT (ML), light greenish gray, wet, very loose, some mica, lacustrine

WELL-GRADED SAND WITH GRAVEL (SM), reddish brown, moist, dense, subangular gravel to 0.6 inches max

POORLY GRADED SAND WITH SILT (SP-SM), white, wet, medium dense

ORGANIC SOIL WITH SAND (OH), dark brown to black, wet, firm to stiff but spongy undisturbed, becomes soft and sticky when remolded, many fine roots, trace of mica

SILTY GRAVEL WITH SAND (GM), brownish red, moist, very dense, subrounded gravel to 1.2 inches max

INTERLAYERED SILT (60 percent) AND CLAY (40 percent): SILT WITH SAND (ML), medium greenish gray, nonplastic, sudden reaction to shaking, layers mostly 1.5 to 8.3 inches thick; LEAN CLAY (CL), dark gray, firm and brittle undisturbed, becomes very soft and sticky when remolded, layers 0.2 to 1.2 inches thick

SILTY SAND WITH GRAVEL (SM), light yellowish brown, moist, medium dense, weak gravel to 1.0 inches max, very few small particles of coal, fill

SANDY ELASTIC SILT (MH), very light gray to white, wet, stiff, weak calcareous cementation

LEAN CLAY WITH SAND (CL/MH), dark brownish gray, moist, stiff

WELL-GRADED GRAVEL WITH SILT (GW-GM), brown, moist, very dense, rounded gravel to 1.0 inches max

Table 2
CRITERIA FOR DESCRIBING MOISTURE CONDITION

| <u>Description</u> | <u>Criteria</u> |
|--------------------|---|
| Dry | Absence of moisture, dusty, dry to the touch |
| Moist | Damp, but no visible water |
| Wet | Visible free water, usually soil is below water table |

Table 3
RELATIVE DENSITY OF COARSE-GRAINED SOIL
(Developed from Sowers, 1979)

| <u>Blows/Ft</u> | <u>Relative Density</u> | <u>Field Test</u> |
|-----------------|-------------------------|---|
| 0-4 | Very loose | Easily penetrated with 1/2-in. steel rod pushed by hand |
| 5-10 | Loose | Easily penetrated with 1/2-in. steel rod pushed by hand |
| 11-30 | Medium | Easily penetrated with 1/2-in. steel rod driven with 5-lb hammer |
| 31-50 | Dense | Penetrated a foot with 1/2-in. steel rod driven with 5-lb hammer |
| >50 | Very dense | Penetrated only a few inches with 1/2-in. steel rod driven with 5-lb hammer |

Table 4
CONSISTENCY OF FINE-GRAINED SOIL
(Developed from Sowers, 1979)

| <u>Blows/Ft</u> | <u>Consistency</u> | <u>Pocket Penetrometer (TSF)</u> | <u>Torvane (TSF)</u> | <u>Field Test</u> |
|-----------------|--------------------|----------------------------------|----------------------|--|
| <2 | Very soft | <0.25 | <0.12 | Easily penetrated several inches by fist |
| 2-4 | Soft | 0.25-0.50 | 0.12-0.25 | Easily penetrated several inches by thumb |
| 5-8 | Firm | 0.50-1.0 | 0.25-0.5 | Can be penetrated several inches by thumb with moderate effort |
| 9-15 | Stiff | 1.0-2.0 | 0.5-1.0 | Readily indented by thumb, but penetrated only with great effort |
| 16-30 | Very stiff | 2.0-4.0 | 1.0-2.0 | Readily indented by thumbnail |
| >30 | Hard | >4.0 | >2.0 | Indented with difficulty by thumbnail |

Locating and Clearing Underground Utilities

I. Purpose

The purpose of this SOP is to provide general guidelines and specific procedures that must be followed on Navy CLEAN projects for locating underground utilities and clearing dig locations in order to maximize our ability to avoid hitting underground utilities and to minimize liabilities to CH2M and its subcontractors and health and safety risks to our project staff.

This SOP shall be used by Activity Managers and Project Managers to, in-turn, develop Activity-specific and project-specific utility location procedures. The activity and project-specific procedures will become part of work plans and project instructions and will be used to prepare scopes of work (SOWs) for the procurement of utility location subcontractors to meet the needs of individual projects.

This SOP also identifies the types of utility locating services that are available from subcontractors and the various tools that are used to locate utilities, and discusses when each type of service and tool may or may not be applicable.

II. Scope

Depending on the Navy/Marine Activity we typically find ourselves in one of two scenarios:

Scenario 1

The Activity provides utility locating (or dig clearance) services through the public works department or similar organization, or has a contract with an outside utility clearance service. Some of these services are provided in the form of dig permits which are required before you can dig or drill. In other cases no official permit is required and the process is somewhat vague.

Scenario 2

The Activity does not get involved in any utility locating processes aside from possibly providing the most recent utility maps, and relies on CH2M to clear the dig locations.

Table 1 provides an up to date summary of which scenarios apply to the various primary Activities served under the Navy CLEAN program.

Scenario 1 is preferred because under this scenario the Navy tends to assume the responsibility if the location is improperly cleared, a utility is struck, and property damage results. However, our experience has been that the clearance services provided by the Navy do not meet the standards that we consider to be adequate, in that they

often simply rely on available base maps to mark utilities and do not verify locations using field geophysics. And if they do use locating tools, they do not provide adequate documentation or marking to confirm that a location has been cleared. So while the Navy's process may protect us from liability for property damage, it does not adequately protect our staff and subcontractors from health risks nor does it compensate us for down time, should a utility be hit.

Therefore, regardless of what services the Navy provides, in most cases we still need to supplement this effort with clearance services from our own third party utility location subcontractor following the procedures and guideline outlined in Section IV of this SOP. The cost implications of providing this service will range from \$500 to several \$1,000 depending on the size of the project.

The scope of services that we ask our subcontractors to provide can involve utility marking/mapping or the clearing of individual dig locations. In the former we ask our subs to mark all utilities within a "site" and often ask them to prepare a map based on their work. In the later, we ask them to clear (identify if there are any utilities within) a certain radius of a proposed dig/drill location.

The appropriate requested scope of services for a project will depend on the project. Clearing individual boreholes is often less expensive and allows the sub to concentrate their efforts on a limited area. However if the scope of the investigation is fluid (all borehole locations are not predetermined) it may be best to mark and map an entire site or keep the subcontractor on call.

Clearance of individual dig locations should be done to a minimum 20 foot radius around the location.

An example SOW for a utility subcontractor procurement is provided in Attachment A.

III. Services and Equipment

This section provides a general description of the services available to help us locate subsurface utilities and describes the types of equipment that these services may (or may not) use to perform their work. It identifies the capabilities of each type of equipment to help the PM specify what they should require from our utility location subs.

Services

The services that are available to us for identifying and marking underground utilities are:

- The local public/private utility-run service such as Miss Utility
- Utility location subcontractors (hired by us)

Attachment B provides a detailed description of each type of organization. It also provides contact numbers and web sites for the various Miss-Utility-type organizations in the areas where we do work for the Navy and contacts and services provided by several subcontractors that we have used or spoken to in the past.

Equipment

Attachment C provides a summary of the various types of equipment used for subsurface utility location. It describes the capabilities and limitations of each in order to help the PM determine if the equipment being used by a subcontractor is adequate.

It is important to make the potential subcontractors aware of the possible types of utilities (and utility materials) that are at the site, and to have them explain in their bid what types of equipment they will use to locate utilities / clear dig locations, and what the limitations of these equipment are.

A list of in-house experts that can be used to help you evaluate bids or answer questions you may have is provided in **Appendix C**.

IV. Procedures and Guidelines

This section presents specific procedures to be followed for the utility location work to be conducted by CH2M and our subcontractors. In addition, a PM will have to follow the procedures required by the Activity to obtain their approvals, clearances and dig permits where necessary. These “dig permit” requirements vary by Activity and must be added to the project-specific SOP, or project instructions. It is preferable that the Activity perform their clearance processes before we follow up with our clearance work.

Activity Notification and Dig Permit Procedures

Identify Activity-specific permit and/or procedural requirements for excavation and drilling activities. Contact the Base Civil Engineer and obtain the appropriate form to begin the clearance process.

Activity Specific: To be provided by Activity or Project Manager

CH2M Utility Clearance Procedures

Do not begin subsurface construction activities (e.g., trenching, excavation, drilling, etc.) until a check for underground utilities and similar obstructions has been conducted by CH2M as a follow-up to the services provided by the Navy. The use of as-built drawings and utility company searches must be supplemented with a geophysical or other survey by a qualified, independent survey contractor (subcontracted to CH2M) to identify additional and undiscovered buried utilities.

Examples of the type of geophysical technologies include (these are further described in Attachment C):

- **Ground Penetrating Radar (GPR)**, which can detect pipes, including gas pipes, tanks, conduits, cables etc, both metallic and non-metallic at depths up to 30 feet depending on equipment. Sensitivity for both minimum object size and maximum depth detectable depends on equipment selected, soil conditions, etc.
- **Radio Frequency (RF)**, involves inducing an RF signal in the pipe or cable and using a receiver to trace it. Some electric and telephone lines emit RF naturally and can be

detected without an induced signal. This method requires knowing where the conductive utility can be accessed to induce RF field if necessary.

- **Dual RF**, a modified version of RF detection using multiple frequencies to enhance sensitivity but with similar limitations to RF
- **Ferromagnetic Detectors**, are metal detectors that will detect ferrous and non-ferrous utilities. Sensitivity is limited, e.g. a 100 mm iron disk to a depth of about one meter or a 25 mm steel paper clip to a depth of about 20 cm.
- **Electronic markers**, are emerging technologies that impart a unique electronic signature to materials such as polyethylene pipe to facilitate location and tracing after installation. Promising for future installations but not of help for most existing utilities already in place.

The following procedures shall be used to identify and mark underground utilities during subsurface construction activities on the project:

- Contact utility companies or the state/regional utility protection service (such as Miss Utility) at least two (2) working days prior to intrusive activities to advise of the proposed work, and ask them to establish the location of the utility underground installations prior to the start of actual excavation: this is a law. These services will only mark the location of public-utility-owned lines and not Navy-owned utilities. In many cases there will not be any public-utility-owned lines on the Activity. There may also be Base-access issues to overcome.
- Procure and schedule the independent survey.
- The survey contractor shall determine the most appropriate geophysical technique or combinations of techniques to identify the buried utilities on the project site, based on the survey contractor's experience and expertise, types of utilities anticipated to be present and specific site conditions. *The types of utilities must be provided to the bidding subcontractors in the SOW and procedures to be used must be specified by the bidder in their bid. It is extremely helpful to provide the sub with utility maps, with the caveat that all utilities are not necessarily depicted.*
- The survey subcontractor shall employ the same geophysical techniques used to identify the buried utilities, to survey the proposed path of subsurface investigation/construction work to confirm no buried utilities are present.
- Obtain utility clearances for subsurface work on both public and private property.
- Clearances provided by both the "Miss Utility" service and the CH2M-subcontracted service are to be in writing, signed by the party conducting the clearance. The Miss Utility service will have standard notification forms/letters which typically simply state that they have been to the site and have done their work. The CH2M subcontractor shall be required to fill out the form provided in Attachment D (this can be modified for a particular project) indicating that each dig/drill location has been addressed. *This documentation requirement (with a copy of the form) needs to be provided in the subcontractor SOW.*

- Marking shall be done using the color coding presented in Attachment E. The type of material used for marking must be approved by the Activity prior to marking. Some base commanders have particular issues with persistent spray paint on their sidewalks and streets. *Any particular marking requirements need to be provided in the subcontractor SOW.*
- Protect and preserve the markings of approximate locations of facilities until the markings are no longer required for safe and proper excavations. If the markings of utility locations are destroyed or removed before excavation commences or is completed, the Project Manager must notify the utility company or utility protection service to inform them that the markings have been destroyed.
- Perform a field check prior to drilling/digging (preferably while the utility location sub is still at the site) to see if field utility markings coincide with locations on utility maps. Look for fire hydrants, valves, manholes, light poles, lighted signs, etc to see if they coincide with utilities identified by the subcontractor.
- Underground utility locations must be physically verified (or dig locations must be physically cleared) by hand digging using wood or fiberglass-handled tools, air knifing, or by some other acceptable means approved by CH2M, when the dig location (e.g. mechanical drilling, excavating) is expected to be within 5 feet of a marked underground system. Hand clearance shall be done to a depth of four feet unless a utility cross-section is available that indicates the utility is at a greater depth. In that event, the hand clearance shall proceed until the documented depth of the utility is reached.
- Conduct a site briefing for employees at the start of the intrusive work regarding the hazards associated with working near the utilities and the means by which the operation will maintain a safe working environment. Detail the method used to isolate the utility and the hazards presented by breaching the isolation.
- Monitor for signs of utilities during advancement of intrusive work (e.g., sudden change in advancement of auger or split spoon during drilling or change in color, texture or density during excavation that could indicate the ground has been previously disturbed).

IV. Attachments

- A- Example SOW for Utility Location Subcontractor Procurement
- B - Services Available for Identifying and Marking Underground Utilities
- C - Equipment Used for Identifying Underground Utilities
- D - Utility Clearance Documentation Form
- E - Utility Marking Color Codes

Attachment A – Example SOW for Subcontracting Underground Utilities Locating Services

CTO-FZ12

Scope of Work

Subsurface Utility Locating

Site XX

**Parcel G Removal Site Evaluation, Former Hunter's Point Naval
Shipyard**

San Francisco, California

A licensed and insured utility locator will be subcontracted to identify and mark out subsurface utilities for an environmental investigation/remediation project at Site XX of Former Hunter's Point Naval Shipyard, San Francisco, California. The subcontractor will need to be available beginning at <<insert time>> on <<insert date>>. It is estimated that the work can be completed within XX days.

Proposed Scope of Work

The subcontractor will identify and mark all subsurface utilities (CHOOSE 1) that lie within a radius of 20 feet of each of XX sampling locations at Site XX shown on the attached Figure 1; (OR) that lie within the bounds of Site XX as delineated on the attached Figure 1. (If multiple sites are to be cleared, provide maps of each site with sample locations or clearance boundaries clearly delineated and a scale provided.)

Utilities will be identified using all reasonably available as-built drawings, electronic locating devices, and any other means necessary to maintain the safety of drilling and sampling personnel and the protection of the base infrastructure. The location of utilities identified from as-built drawings or other maps must be verified in the field prior to marking.

Base utility drawings for the Site(s) (CHOOSE 1) can be found at <<insert specific department and address or phone number on the base>> and should be reviewed by the subcontractor and referenced as part of the utility locating. (OR), will be provided to the subcontractor by CH2M HILL upon the award of the subcontract. (OR), are not available. Utility drawings shall not be considered definitive and must be field verified.

Field verification will include detection using nonintrusive subsurface detection equipment (magnetometers, GPR, etc) as well as opening manhole covers to verify pipe directions. As part of the bid, the Subcontractor shall provide a list of the various subsurface investigation tools they propose to have available and use at the site and what the limitations are of each tool.

A CH2M HILL representative shall be present to coordinate utility clearance activities and identify points and features to be cleared.

Field Marking and Documentation

All utilities located within **(CHOOSE 1) a 20-ft radius of the XX proposed soil boring locations (OR) within the boundary of the site(s)** as identified on the attached figure(s) will be marked using **paint (some Bases such as the WNY may have restrictions on the use of permanent paint)** and/or pin flags color coded to indicate electricity, gas, water, steam, telephone, TV cable, fiber optic, sewer, etc. The color coding shall match the industry standard as described on the attached form. In addition, the **Buried Utility Location Tracking Form** (attached) will be completed by the Subcontractor based upon what is identified in the field during the utility locating and submitted back to CH2M HILL (field staff or project manager) within 24 hours of completing the utility locating activities.

(OPTIONAL) *The subcontractor shall also provide a map (or hand sketch) of the identified utilities to the Engineer within XX days of field demobilization. The map shall include coordinates or ties from fixed surface features to each identified subsurface utility.*

Bid Sheet/Payment Units

The subcontractor will bid on a time and materials basis for time spent on site and researching utility maps. Mobilization (including daily travel to the site) should be bid as a lump sum, as well as the preparation of the AHA and any required mapping. The per diem line item should be used if the field crew will require overnight accommodations at the project site.

Health and Safety Requirements

The utility locating subcontractor is to provide and assume responsibility for an adequate corporate Health and Safety Plan for onsite personnel. Standard personal safety equipment including: hard hat, safety glasses, steel-toed boots, gloves are recommended for all project activities. Specific health and safety requirements will be established by the Subcontractor for each project. The health and safety requirements will be subject to the review of CH2M HILL.

The subcontractor shall also prepare and provide to the Engineer, at least 48 hours prior to mobilization, an acceptable Activity Hazard Analysis (AHA) using the attached AHA form or similar.

It is also required that all subcontractor personnel who will be on site attend the daily 15-minute health and safety tailgate meeting at the start of each day in the field.

Subcontractor personnel showing indications of being under the influence of alcohol or illegal drugs will be sent off the job site and their employers will be notified. Subcontractor personnel under the influence of prescription or over-the-counter medication that may impair their ability to operate equipment will not be permitted to do so. It is expected that the subcontractor will assign them other work and provide a capable replacement (if necessary) to operate the equipment to continue work.

Security

The work will be performed on US Navy property. CH2M HILL will identify the Subcontractor personnel who will perform the work to the appropriate Navy facility point-of-contact, and will identify the Navy point-of-contact to the Subcontractor crew. The Subcontractor bears final responsibility for coordinating access of his personnel onto Navy property to perform required work. This responsibility includes arranging logistics and providing to CH2M HILL, in advance or at time of entry as specified, any required identification information for the Subcontractor personnel. Specifically, the following information should be submitted with the bid package for all personnel that will perform the work in question (this information is required to obtain a base pass):

- Name
- Birth Place
- Birth Date
- Social Security Number
- Drivers License State and Number
- Citizenship

Please be advised that no weapons, alcohol, or drugs will be permitted on the Navy facility at any time. If any such items are found, they will be confiscated, and the Subcontractor will be dismissed.

Quality Assurance

The Subcontractor will be licensed and insured to operate in the State of California and will comply with all applicable federal, state, county and local laws and regulations. The subcontractor will maintain, calibrate, and operate all electronic locating instruments in accordance with the manufacturer's recommendations. Additionally, the Subcontractor shall make all reasonable efforts to review as-built engineering drawings maintained by Base personnel, and shall notify the CH2M HILL Project Manager in writing (email is acceptable) whenever such documentation was not available or could not be reviewed.

Subcontractor Standby Time

At certain periods during the utility locating activities, the Subcontractor's personnel may be asked to stop work and standby when work may normally occur. During such times, the Subcontractor will cease activities until directed by the CH2M HILL representative to resume operations. Subcontractor standby time also will include potential delays caused by the CH2M HILL representative not arriving at the site by the agreed-upon meeting time for start of the work day. Standby will be paid to the

Subcontractor at the hourly rate specified in the Subcontractor's Bid Form attached to these specifications.

Cumulative Subcontractor standby will be accrued in increments no shorter than 15 minutes (i.e., an individual standby episode of less than 15 minutes is not chargeable).

During periods for which standby time is paid, the surveying equipment will not be demobilized and the team will remain at the site. At the conclusion of each day, the daily logs for the Subcontractor and CH2M HILL representative will indicate the amount of standby time incurred by the Subcontractor, if any. Payment will be made only for standby time recorded on CH2M HILL's daily logs.

Down Time

Should equipment furnished by the Subcontractor malfunction, preventing the effective and efficient prosecution of the work, or inclement weather conditions prevent safe and effective work from occurring, down time will be indicated in the Subcontractor's and CH2M Hill representative's daily logs. No payment will be made for down time.

Schedule

It is anticipated that the subsurface utility locating activities will occur on <<insert date>>. It is estimated that the above scope will be completed within XXX days.

Attachment B - Services Available for Identifying and Marking Underground Utilities

The services that are available to us for identifying and marking underground utilities are:

- The Activity's PWC (or similar organization)
- The local public/private utility -run service such as Miss Utility
- Utility location subcontractors (hired by CH2M HILL)

Each are discussed below.

Navy Public Works Department

A Public Works Department (PWD) is usually present at each Activity. The PWD is responsible for maintaining the public works at the base including management of utilities. In many cases, the PWD has a written permit process in place to identify and mark-out the locations of Navy-owned utilities [Note: The PWD is usually NOT responsible for the locations/mark-outs of non-Navy owned, public utilities (e.g., Washington Gas, Virginia Power, municipal water and sewer, etc.). Therefore, it is likely that we will have to contact other organizations besides the PWD in order to identify non-Navy owned, public utilities].

At some Activities, there may not be a PWD, the PWD may not have a written permit process in place, or the PWD may not take responsibility for utility locating and mark-outs. In these cases, the PWD should still be contacted since it is likely that they will have the best understanding of the utility locations at the Activity (i.e., engineering drawings, institutional knowledge, etc.). Subsequently, the PWD should be brought into a cooperative arrangement (if possible) with the other services employed in utility locating and mark-out in order to have the most comprehensive assessment performed.

At all Activities we should have a contact (name and phone number), and preferably an established relationship, with PWD, either directly or through the NAVFAC Atlantic, Midlant, or Washington NTR or Activity Environmental Office that we can work with and contact in the event of problems.

Miss Utility or "One Call" Services for Public Utility Mark-outs

Miss Utility or "One Call" service centers are information exchange centers for excavators, contractors and property owners planning any kind of excavation or digging. The "One Call" center notifies participating public utilities of the upcoming excavation work so they can locate and mark their underground utilities in advance to prevent possible damage to underground utility lines, injury, property damage and service outages. In some instances, such with southeastern Virginia bases, the Navy has entered into agreement with Ms. Utilities and is part of the response process for Miss

Utilities. Generally, a minimum of 48 hours is required for the public utility mark-outs to be performed. The "One Call" services are free to the public. Note that the "One Call" centers only coordinate with participating public utilities. There may be some public utilities that do NOT participate in the "One Call" center which may need to be contacted separately. For example, in Washington, DC, the Miss Utility "One Call" center does not locate and mark public sewer and water lines. Therefore, the municipal water and sewer authority must be contacted separately to have the sewer and water lines marked out. The AM should contact the appropriate one-call center to determine their scope of services.

For the Mid-Atlantic region, the following "One Call" service centers are available.

| Name | Phone | Website | Comments |
|--|------------------------------|--|--|
| Miss Utility of DELMARVA | 800-257-7777 | www.missutility.net | Public utility mark-outs in Delaware, Maryland, Washington, DC, and Northern Virginia |
| Miss Utility of Southern Virginia (One Call) | 800-552-7001 | not available | Public utility mark-outs in Southern Virginia |
| Miss Utility of Virginia | 800-257-7777 800-552-7007 | www.missutilityofvirginia.com | General information on public utility mark-outs in Virginia, with links to Miss Utility of DELMARVA and Miss Utility of Southern Virginia (One Call) |
| Miss Utility of West Virginia, Inc | 800-245-4848 | none | Call to determine what utilities they work with in West Virginia |
| North Carolina One Call Center | 800-632-4949 | www.ncocc.org/ncocc/default.htm | Public Utility Markouts in North Carolina |

Private Subcontractors

- Utility-locating support is required at some level for most all CH2M HILL field projects in "clearing" proposed subsurface boring locations on the project site. Utility location and sample clearance can include a comprehensive effort of GIS map interpretation, professional land surveying, field locating, and geophysical surveying. Since we can usually provide our own GIS-related services for projects and our professional land surveying services are normally procured separately, utility-locating subcontractors will normally only be required for some level of geophysical surveying support in the field. This level of geophysical surveying support can range widely from a simple electromagnetic (EM) survey over a known utility line, to a blind geophysical effort, including a ground-penetrating radar (GPR) survey and/or a comprehensive EM survey to delineate and characterize all unknown subsurface anomalies.

The level of service required from the subcontractor will vary depending on the nature of the site. At sites where utility locations are well defined on the maps and recent construction is limited, CH2M HILL may be confident with a limited effort from a traditional utility-locating subcontractor providing a simple EM survey. At

sites where utility locations are not well defined, where recent constructions may have altered utility locations, or the nature of the site makes utility location difficult, CH2M HILL will require the services of a comprehensive geophysical surveying subcontractor, with a wide range of GPR and EM services available for use on an "as-needed" basis. Typical costs for geophysical surveying subcontractors will range from approximately \$200 per day for a simple EM effort (usually one crew member and one instrument) to approximately \$1,500 per day for a comprehensive geophysical surveying effort (usually a two-person crew and multiple instruments). Comprehensive geophysical surveying efforts may also include field data interpretation (and subsequent report preparation) and non-destructive excavation to field-verify utility depths and locations.

The following table provides a list of recommended geophysical surveying support subcontractors that can be used for utility-locating services:

| Company Name and Address | Contact Name and Phone Number | Equipment ¹ | | | | | Other Services ² | | |
|--|---------------------------------|------------------------|---|---|---|---|-----------------------------|---|---|
| | | 1 | 2 | 3 | 4 | 5 | A | B | C |
| US Radar, Inc.* PO Box 319 Matawan, NJ 07747 | Ron LaBarca 732-566-2035 | | | 4 | | | | | |
| Utilities Search, Inc.* | Jim Davis 703-369-5758 | 4 | | | | 4 | 4 | 4 | 4 |
| So Deep, Inc.* 8397 Euclid Avenue Manassas Park, VA 20111 | 703-361-6005 | 4 | | | | | 4 | 4 | 4 |
| Accurate Locating, Inc. 1327 Ashton Rd., Suite 101 Hanover, MD 21076 | Ken Shipley 410-850-0280 | 4 | 4 | | | | | | |
| NAEVA Geophysics, Inc. P.O. Box 7325 Charlottesville, VA 22906 | Alan Mazurowski 434-978-3187 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 |
| Earth Resources Technology, Inc. 8106 Stayton Rd. Jessup, MD 20794 | Peter Li 240-554-0161 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | |
| Geophex, Ltd 605 Mercury Street Raleigh, NC 27603 | I. J. Won 919-839-8515 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 |
| | | | | | | | | | |

Notes:

*Companies denoted with an asterisk have demonstrated reluctance to assume responsibility for damage to underground utilities or an inability to accommodate the insurance requirements that CH2M HILL requests for this type of work at many Navy sites.

¹Equipment types are:

1. Simple electromagnetic instruments, usually hand-held
2. Other, more innovative, electromagnetic instruments, including larger instruments for more area coverage
3. Ground-penetrating radar systems of all kinds
4. Audio-frequency detectors of all kinds
5. Radio-frequency detectors of all kinds

²Other services include:

- A. Data interpretation and/or report preparation to provide a permanent record of the geophysical survey results and a professional interpretation of the findings, including expected accuracy and precision.
 - B. Non-destructive excavation to field-verify the depths, locations, and types of subsurface utilities.
 - C. Concrete/asphalt coring and pavement/surface restoration.
-

Attachment C – Equipment Used for Identifying Underground Utilities

This attachment provides a summary of the various types of equipment used for subsurface utility location. It describes the capabilities and limitations of each in order to help the AM and PM determine if the equipment being proposed by a subcontractor or Navy is adequate. A list of in-house experts that can be used to answer questions you may have is provided below.

CH2M HILL In-house Utility Location Experts

Tamir Klaff/WDC

Home Office Phone – 703-669-9611

Electromagnetic Induction (EMI) Methods

EMI instruments, in general, induce an electromagnetic field into the ground (the primary field) and then record the response (the secondary field), if any. Lateral changes in subsurface conductivity, such as caused by the presence of buried metal or by significant soil variations, cause changes in the secondary field recorded by the instrument and thus enable detection and mapping of the subsurface features. It should be noted that EMI only works for electrically conductive materials--plastic or PVC pipes are generally not detected with EMI. Water and gas lines are commonly plastic, although most new lines include a copper “locator” strip on the top of the PVC to allow for detection with EMI.

EMI technology encompasses a wide range of instruments, each with inherent strengths and weaknesses for particular applications. One major division of EMI is between “time-domain” and “frequency-domain” instruments that differ in the aspect of the secondary field they detect. Another difference in EMI instruments is the operating frequency they use to transmit the primary field. Audio- and radio-frequencies are often used for utility detection, although other frequencies are also used. Consideration of the type of utility expected, surface features that could interfere with detection, and the “congestion” of utilities in an area, should be made when choosing a particular EMI instrument for a particular site.

One common EMI tool used for utility location is a handheld unit that can be used to quickly scan an area for utilities and allows for marking locations in “real time”. This method is most commonly used by “dig-safe” contractors marking out known utilities prior to excavation. It should be noted that this method works best when a signal (the primary field) can be placed directly onto the line (i.e., by clamping or otherwise connecting to the end of the line visible at the surface, or for larger utilities such as sewers, by running a transmitter through the utility). These types of tools also have a limited capability to scan an area for unknown utilities. Usually this requires having enough area to separate a hand held transmitter at least a hundred feet from the

receiver. Whether hunting for unknown, or confirming known, utilities, this method will only detect continuous lengths of metallic conductors.

In addition to the handheld EMI units, larger, more powerful EMI tools are available that provide more comprehensive detection and mapping of subsurface features. Generally, data with these methods are collected on a regular grid in the investigation area, and are then analyzed to locate linear anomalies that can be interpreted as utilities. These methods will usually detect *all* subsurface metal (above a minimum size), including pieces of abandoned utilities. In addition, in some situations, backfill can be detected against native soils giving information on trenching and possible utility location. Drawbacks to these methods are that the secondary signals from utilities are often swamped (i.e., undetectable) close to buildings and other cultural features, and that the subsurface at heavily built-up sites may be too complicated to confidently interpret completely.

Hand-held metal detectors (treasure-finders) are usually based on EMI technology. They can be used to locate shallow buried metal associated with utilities (e.g., junctions, manholes, metallic locators). Advantages of these tools is the ease of use and real-time marking of anomalies. Drawbacks include limited depths of investigations and no data storage capacity.

Ground Penetrating Radar (GPR)

GPR systems transmit radio and microwave frequency (e.g., 80 megaHertz to 1,000 megaHertz) waves into the ground and then record reflections of those waves coming back to the surface. Reflections of the radar waves typically occur at lithologic changes, subsurface discontinuities, and subsurface structures. Plastic and PVC pipes can sometimes be detected in GPR data, especially if they are shallow, large, and full of a contrasting material such as air in a wet soil, or water in a dry soil. GPR data are usually collected in regular patterns over an area and then analyzed for linear anomalies that can be interpreted as utilities. GPR is usually very accurate in x-y location of utilities, and can be calibrated at a site to give very accurate depth information as well. A significant drawback to GPR is that depth of investigation is highly dependant on background soil conductivity, and it will not work on all sites. It is not uncommon to get only 1-2 feet of penetration with the signal in damp, clayey environments. Another drawback to GPR is that sites containing significant fill material (e.g., concrete rubble, scrap metal, garbage) will result in complicated anomalies that are difficult or impossible to interpret.

Magnetic Field Methods

Magnetic field methods rely on detecting changes to the earth's magnetic field caused by ferrous metal objects. This method is usually more sensitive to magnetic metal (i.e., deeper detection) than EMI methods. A drawback to this method is it is more susceptible to being swamped by surface features such as fences and cars. In addition, procedures must usually be implemented that account for natural variations in the earth's background field as it changes throughout the day. One common use of the method is to measure and analyze the gradient of the magnetic field, which eliminates most of the drawbacks to the method. It should be noted this method only detects

ferrous metal, primarily iron and steel for utility location applications. Some utility detector combine magnetic and EMI methods into a single hand-held unit.

Optical Methods

Down the hole cameras may be useful in visually reviewing a pipe for empty conduits and/or vaults.

Attachment D – Utility Clearance Documentation Form

Attachment E – Utility Marking Color Codes

The following is the standard color code used by industry to mark various types of utilities and other features at a construction site.

White – Proposed excavations and borings

Pink – Temporary survey markings

Red – Electrical power lines, cables, conduits and lighting cables

Yellow – Gas, oil, steam, petroleum or gaseous materials

Orange – Communication, alarm or signal lines, cables, or conduits

Blue – Potable water

Purple – Reclaimed water, irrigation and slurry lines

Green – Sewer and storm drain lines

Decontamination of Personnel and Equipment

I. Purpose

To provide general guidelines for the decontamination of personnel, sampling equipment, and monitoring equipment used in potentially contaminated environments.

II. Scope

This is a general description of decontamination procedures.

III. Equipment and Materials

- Demonstrated analyte-free, deionized ("DI") water (specifically, ASTM Type II water or lab-grade DI water)
- Potable water; must be from a municipal water supplier, otherwise an analysis must be run for appropriate volatile and semivolatile organic compounds and inorganic chemicals (e.g., Target Compound List and Target Analyte List chemicals)
- 2.5% (W/W) Liquinox[®] and water solution
- Concentrated (V/V) pesticide grade isopropanol (DO NOT USE ACETONE)
- Large plastic pails or tubs for Liquinox[®] and water, scrub brushes, squirt bottles for Liquinox[®] solution, methanol and water, plastic bags and sheets
- DOT approved 55-gallon drum for disposal of waste
- Personal Protective Equipment as specified by the Health and Safety Plan
- Decontamination pad and steam cleaner/high pressure cleaner for large equipment

IV. Procedures and Guidelines

A. PERSONNEL DECONTAMINATION

To be performed after completion of tasks whenever potential for contamination exists, and upon leaving the exclusion zone.

1. Wash boots in Liquinox[®] solution, then rinse with water. If disposable latex booties are worn over boots in the work area, rinse with Liquinox[®] solution, remove, and discard into DOT-approved 55-gallon drum.
2. Wash outer gloves in Liquinox[®] solution, rinse, remove, and discard into DOT-approved 55-gallon drum.
3. Remove disposable coveralls ("Tyveks") and discard into DOT-approved 55-gallon drum.
4. Remove respirator (if worn).
5. Remove inner gloves and discard.
6. At the end of the work day, shower entire body, including hair, either at the work site or at home.
7. Sanitize respirator if worn.

B. SAMPLING EQUIPMENT DECONTAMINATION – GROUNDWATER SAMPLING PUMPS

Sampling pumps are decontaminated after each use as follows.

1. Don phthalate-free gloves.
2. Spread plastic on the ground to keep equipment from touching the ground
3. Turn off pump after sampling. Remove pump from well and remove and dispose of tubing. Place pump in decontamination tube.
4. Turn pump back on and pump 1 gallon of Liquinox[®] solution through the sampling pump.
5. Rinse with 1 gallon of 10% isopropanol solution pumped through the pump. (DO NOT USE ACETONE). (Optional)
6. Rinse with 1 gallon of tap water.
7. Rinse with 1 gallon of deionized water.
8. Keep decontaminated pump in decontamination tube or remove and wrap in aluminum foil or clean plastic sheeting.
9. Collect all rinsate and dispose of in a DOT-approved 55-gallon drum.
10. Decontamination materials (e.g., plastic sheeting, tubing, etc.) that have come in contact with used decontamination fluids or sampling equipment will be disposed of in either DOT-approved 55-gallon drums or with solid waste in garbage bags, dependent on Facility/project requirements.

C. SAMPLING EQUIPMENT DECONTAMINATION – OTHER EQUIPMENT

Reusable sampling equipment is decontaminated after each use as follows.

1. Don phthalate-free gloves.
2. Before entering the potentially contaminated zone, wrap soil contact points in aluminum foil (shiny side out).
3. Rinse and scrub with potable water.
4. Wash all equipment surfaces that contacted the potentially contaminated soil/water with Liquinox[®] solution.
5. Rinse with potable water.
6. Rinse with distilled or potable water and isopropanol solution (DO NOT USE ACETONE). (Optional)
7. Air dry.
8. Rinse with deionized water.
9. Completely air dry and wrap exposed areas with aluminum foil (shiny side out) for transport and handling if equipment will not be used immediately.
10. Collect all rinsate and dispose of in a DOT-approved 55-gallon drum.
11. Decontamination materials (e.g., plastic sheeting, tubing, etc.) that have come in contact with used decontamination fluids or sampling equipment will be disposed of in DOT-approved 55-gallon drums or with solid waste in garbage bags, dependent on Facility/project requirements.

D. HEALTH AND SAFETY MONITORING EQUIPMENT DECONTAMINATION

1. Before use, wrap soil contact points in plastic to reduce need for subsequent cleaning.
2. Wipe all surfaces that had possible contact with contaminated materials with a paper towel wet with Liquinox[®] solution, then a towel wet with methanol solution, and finally three times with a towel wet with distilled water. Dispose of all used paper towels in a DOT-approved 55-gallon drum or with solid waste in garbage bags, dependent on Facility/project requirements.

E. SAMPLE CONTAINER DECONTAMINATION

The outsides of sample bottles or containers filled in the field may need to be decontaminated before being packed for shipment or handled by personnel without hand protection. The procedure is:

1. Wipe container with a paper towel dampened with Liquinox® solution or immerse in the solution AFTER THE CONTAINERS HAVE BEEN SEALED. Repeat the above steps using potable water.
2. Dispose of all used paper towels in a DOT-approved 55-gallon drum or with solid waste in garbage bags, dependent on Facility/project requirements.

F. HEAVY EQUIPMENT AND TOOLS

Heavy equipment such as drilling rigs, drilling rods/tools, and the backhoe will be decontaminated upon arrival at the site and between locations as follows:

1. Set up a decontamination pad in area designated by the Facility
2. Steam clean heavy equipment until no visible signs of dirt are observed. This may require wire or stiff brushes to dislodge dirt from some areas.

V. Attachments

None.

VI. Key Checks and Items

- Clean with solutions of Liquinox®, Liquinox® solution (optional), and distilled water.
- Do not use acetone for decontamination.
- Drum all contaminated rinsate and materials.
- Decontaminate filled sample bottles before relinquishing them to anyone.

Preparing Field Log Books

I. Purpose

This SOP provides general guidelines for entering field data into log books during site investigation and remediation activities.

II. Scope

This is a general description of data requirements and format for field log books. Log books are needed to properly document all field activities in support of data evaluation and possible legal activities.

III. Equipment and Materials

- Log book
- Indelible pen

IV. Procedures and Guidelines

Properly completed field log books are a requirement for much of the work we perform under the Navy CLEAN contract. Log books are legal documents and, as such, must be prepared following specific procedures and must contain required information to ensure their integrity and legitimacy. This SOP describes the basic requirements for field log book entries.

A. PROCEDURES FOR COMPLETING FIELD LOG BOOKS

1. Field notes commonly are kept in bound, hard-cover logbooks used by surveyors and produced, for example, by Peninsular Publishing Company and SESCO, Inc. Pages should be water-resistant and notes should be taken only with water-proof, non-erasable permanent ink, such as that provided in Sanford Sharpie® permanent markers. **Note:** for sites where PFC is being analyzed for, Rite-in-the-Rain®, Sanford Sharpie®, or anything water-resistant or with Teflon® cannot be used in the field. All field book materials must be “fluorine free”. Acceptable substitutes would be a sewn notebook without a plastic cover, or loose leaf notebook paper.
2. On the inside cover of the log book the following information should be included:

- Company name and address
 - Log-holders name if log book was assigned specifically to that person
 - Activity or location
 - Project name
 - Project manager's name
 - Phone numbers of the company, supervisors, emergency response, etc.
3. All lines of all pages should be used to prevent later additions of text, which could later be questioned. Any line not used should be marked through with a line and initialed and dated. Any pages not used should be marked through with a line, the author's initials, the date, and the note "Intentionally Left Blank."
 4. If errors are made in the log book, cross a single line through the error and enter the correct information. All corrections shall be initialed and dated by the personnel performing the correction. If possible, all corrections should be made by the individual who made the error.
 5. Daily entries will be made chronologically.
 6. Information will be recorded directly in the field log book during the work activity. Information will not be written on a separate sheet and then later transcribed into the log book.
 7. Each page of the log book will have the date of the work and the note takers initials.
 8. The final page of each day's notes will include the note-takers signature as well as the date.
 9. Only information relevant to the subject project will be added to the log book.
 10. The field notes will be copied and the copies sent to the Project Manager or designee in a timely manner (at least by the end of each week of work being performed).

B. INFORMATION TO BE INCLUDED IN FIELD LOG BOOKS

1. Entries into the log book should be as detailed and descriptive as possible so that a particular situation can be recalled without reliance on the collector's memory. Entries must be legible and complete.
2. General project information will be recorded at the beginning of each field project. This will include the project title, the project number, and project staff.

3. Scope: Describe the general scope of work to be performed each day.
4. Weather: Record the weather conditions and any significant changes in the weather during the day.
5. Tail Gate Safety Meetings: Record time and location of meeting, who was present, topics discussed, issues/problems/concerns identified, and corrective actions or adjustments made to address concerns/problems, and other pertinent information.
6. Standard Health and Safety Procedures: Record level of personal protection being used (e.g., level D PPE), record air monitoring data on a regular basis and note where data were recording (e.g., reading in borehole, reading in breathing zone, etc). Also record other required health and safety procedures as specified in the project specific health and safety plan.
7. Instrument Calibration; Record calibration information for each piece of health and safety and field equipment.
8. Personnel: Record names of all personnel present during field activities and list their roles and their affiliation. Record when personnel and visitors enter and leave a project site and their level of personal protection.
9. Communications: Record communications with project manager, subcontractors, regulators, facility personnel, and others that impact performance of the project.
10. Time: Keep a running time log explaining field activities as they occur chronologically throughout the day.
11. Deviations from the Work Plan: Record any deviations from the work plan and document why these were required and any communications authorizing these deviations.
12. Heath and Safety Incidents: Record any health and safety incidents and immediately report any incidents to the Project Manager.
13. Subcontractor Information: Record name of company, record names and roles of subcontractor personnel, list type of equipment being used and general scope of work. List times of starting and stopping work and quantities of consumable equipment used if it is to be billed to the project.
14. Problems and Corrective Actions: Clearly describe any problems encountered during the field work and the corrective actions taken to address these problems.
15. Technical and Project Information: Describe the details of the work being performed. The technical information recorded will vary significantly between projects. The project work plan will describe the specific activities to be performed and may also list requirements

for note taking. Discuss note-taking expectations with the Project Manager prior to beginning the field work.

16. Any conditions that might adversely affect the work or any data obtained (e.g., nearby construction that might have introduced excessive amounts of dust into the air).
17. Sampling Information; Specific information that will be relevant to most sampling jobs includes the following:
 - Description of the general sampling area – site name, buildings and streets in the area, etc.
 - Station/Location identifier
 - Description of the sample location – estimate location in comparison to two fixed points – draw a diagram in the field log book indicating sample location relative to these fixed points – include distances in feet.
 - Sample matrix and type
 - Sample date and time
 - Sample identifier
 - Draw a box around the sample ID so that it stands out in the field notes
 - Information on how the sample was collected – distinguish between “grab,” “composite,” and “discrete” samples
 - Number and type of sample containers collected
 - Record of any field measurements taken (i.e. pH, turbidity, dissolved oxygen, and temperature, and conductivity)
 - Parameters to be analyzed for, if appropriate
 - Descriptions of soil samples and drilling cuttings can be entered in depth sequence, along with PID readings and other observations. Include any unusual appearances of the samples.

C. SUGGESTED FORMAT FOR RECORDING FIELD DATA

1. Use the left side border to record times and the remainder of the page to record information (see attached example).
2. Use tables to record sampling information and field data from multiple samples.
3. Sketch sampling locations and other pertinent information.
4. Sketch well construction diagrams.

V. Attachments

Example field notes.

Chain-of-Custody

I Purpose

The purpose of this SOP is to provide information on chain-of-custody procedures to be used under the CLEAN Program.

II Scope

This procedure describes the steps necessary for transferring samples through the use of Chain-of-Custody Records. A Chain-of-Custody Record is required, without exception, for the tracking and recording of samples collected for on-site or off-site analysis (chemical or geotechnical) during program activities (except wellhead samples taken for measurement of field parameters). Use of the Chain-of-Custody Record Form creates an accurate written record that can be used to trace the possession and handling of the sample from the moment of its collection through analysis. This procedure identifies the necessary custody records and describes their completion. This procedure does not take precedence over region specific or site-specific requirements for chain-of-custody.

III Definitions

Chain-of-Custody Record Form - A Chain-of-Custody Record Form is a printed two-part form that accompanies a sample or group of samples as custody of the sample(s) is transferred from one custodian to another custodian. One copy of the form must be retained in the project file.

Custodian - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under one's custody if:

- It is in one's actual possession.
- It is in one's view, after being in one's physical possession.
- It was in one's physical possession and then he/she locked it up to prevent tampering.
- It is in a designated and identified secure area.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

IV. Procedures

The term “chain-of-custody” refers to procedures which ensure that evidence presented in a court of law is valid. The chain-of-custody procedures track the evidence from the time and place it is first obtained to the courtroom, as well as providing security for the evidence as it is moved and/or passed from the custody of one individual to another.

Chain-of-custody procedures, recordkeeping, and documentation are an important part of the management control of samples. Regulatory agencies must be able to provide the chain-of-possession and custody of any samples that are offered for evidence, or that form the basis of analytical test results introduced as evidence. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

Sample Identification

The method of identification of a sample depends on the type of measurement or analysis performed. When *in situ* measurements are made, the data are recorded directly in bound logbooks or other field data records with identifying information.

Information which shall be recorded in the field logbook, when in-situ measurements or samples for laboratory analysis are collected, includes:

- Field Sampler(s),
- Contract Task Order (CTO) Number,
- Project Sample Number,
- Sample location or sampling station number,
- Date and time of sample collection and/or measurement,
- Field observations,
- Equipment used to collect samples and measurements, and
- Calibration data for equipment used

Measurements and observations shall be recorded using waterproof ink.

Sample Label

Samples, other than for *in situ* measurements, are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often divided into portions, depending upon the analyses to be performed. Each portion is preserved in accordance with the Sampling and Analysis Plan. Each sample container is identified by a sample label (see Attachment A). Sample labels are provided, along with sample containers, by the analytical laboratory. The information recorded on the sample label includes:

- Project - CTO Number.
- Station Location - The unique sample number identifying this sample.
- Date - A six-digit number indicating the day, month, and year of sample collection (e.g., 05/21/17).

- Time - A four-digit number indicating the 24-hour time of collection (for example: 0954 is 9:54 a.m., and 1629 is 4:29 p.m.).
- Medium - Water, soil, sediment, sludge, waste, etc.
- Sample Type - Grab or composite.
- Preservation - Type and quantity of preservation added.
- Analysis - VOA, BNAs, PCBs, pesticides, metals, cyanide, other.
- Sampled By - Printed name of the sampler.
- Remarks - Any pertinent additional information.

Using only the work assignment number of the sample label maintains the anonymity of sites. This may be necessary, even to the extent of preventing the laboratory performing the analysis from knowing the identity of the site (e.g., if the laboratory is part of an organization that has performed previous work on the site). The field team should always follow the sample ID system prepared by the project EIS and reviewed by the Project Manager.

Chain-of-Custody Procedures

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures until it is in the custody of the analytical laboratory and has been stored or disposed.

Field Custody Procedures

- Samples are collected as described in the site Sampling and Analysis Plan. Care must be taken to record precisely the sample location and to ensure that the sample number on the label matches the Chain-of-Custody Record exactly.
- A Chain-of-Custody Record will be prepared for each individual cooler shipped and will include *only* the samples contained within that particular cooler. The Chain-of-Custody Record for that cooler will then be sealed in a zip-log bag and placed in the cooler prior to sealing. This ensures that the laboratory properly attributes trip blanks with the correct cooler and allows for easier tracking should a cooler become lost during transit.
- The person undertaking the actual sampling in the field is responsible for the care and custody of the samples collected until they are properly transferred or dispatched.
- When photographs are taken of the sampling as part of the documentation procedure, the name of the photographer, date, time, site location, and site description are entered sequentially in the site logbook as photos are taken. Once downloaded to the server or developed, the electronic files or photographic prints shall be serially numbered, corresponding to the logbook descriptions; photographic prints will be stored in the project files. To identify

sample locations in photographs, an easily read sign with the appropriate sample location number should be included.

- Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions (e.g., a logbook notation would explain that a pencil was used to fill out the sample label if the pen would not function in freezing weather.)

Transfer of Custody and Shipment

Samples are accompanied by a Chain-of-Custody Record Form. **A Chain-of-Custody Record Form must be completed for each cooler and should include only the samples contained within that cooler.** A Chain-of-Custody Record Form example is shown in Attachment B. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the Record. This Record documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory. The Chain-of-Custody Record is filled out as given below:

- Enter header information (CTO number, samplers, and project name).
- Enter sample specific information (sample number, media, sample analysis required and analytical method grab or composite, number and type of sample containers, and date/time sample was collected).
- Sign, date, and enter the time under “Relinquished by” entry.
- Have the person receiving the sample sign the “Received by” entry. If shipping samples by a common carrier, print the carrier to be used in this space (i.e., Federal Express).
- If a carrier is used, enter the airbill number under “Remarks,” in the bottom right corner;
- Place the original (top, signed copy) of the Chain-of-Custody Record Form in a plastic zipper-type bag or other appropriate sample-shipping package. Retain the copy with field records.
- Sign and date the custody seal, a 1-inch by 3-inch white paper label with black lettering and an adhesive backing. Attachment C is an example of a custody seal. The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field. Custody seals shall be provided by the analytical laboratory.
- Place the seal across the shipping container opening (front and back) so that it would be broken if the container were to be opened.
- Complete other carrier-required shipping papers.

The custody record is completed using waterproof ink. Any corrections are made by drawing a line through and initialing and dating the change, then entering the correct information. Erasures are not permitted.

Common carriers will usually not accept responsibility for handling Chain-of-Custody Record Forms; this necessitates packing the record in the shipping container (enclosed with other documentation in a plastic zipper-type bag). As long as custody forms are sealed inside the shipping container and the custody seals are intact, commercial carriers are not required to sign the custody form.

The laboratory representative who accepts the incoming sample shipment signs and dates the Chain-of-Custody Record, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal logbooks and custody records throughout sample preparation and analysis.

V Quality Assurance Records

Once samples have been packaged and shipped, the Chain-of-Custody copy and airbill receipt become part of the quality assurance record.


VI Attachments

- A. Sample Label
- B. Chain of Custody Form
- C. Custody Seal

VII References

USEPA. *User's Guide to the Contract Laboratory Program*. Office of Emergency and Remedial Response, Washington, D.C. (EPA/540/P-91/002), January 1991.

Attachment A
Example Sample Label

| | |
|--|---|
| |  Quality Analytical Laboratories, Inc. 2567 Fairlane Drive Montgomery, Alabama 36116 PH. (334)271-2440 |
| | Client _____ |
| | Sample No. _____ |
| | Location _____ |
| | Analysis _____ |
| | Preservative HCL _____ |
| | Date _____ By _____ |

| | |
|---|--------------|
| CEIMIC CORPORATION 18 Dean Knapp Drive, Narragansett, R.I. 02882 • (401) 752-8000 | |
| SITE NAME | DATE |
| ANALYSIS | TIME |
| | PRESERVATIVE |
| SAMPLE TYPE | |
| <input type="checkbox"/> Grab <input type="checkbox"/> Composite <input type="checkbox"/> Other _____ | |
| COLLECTED BY: | |

Attachment B
Example Chain-of-Custody Record

DISTRIBUTION: Original - LAB, Yellow - LAB, Pink - Client
REV 3/94 FORM 340

Attachment C
Example Custody Seal



CUSTODY SEAL

Date

Signature

Packaging and Shipping Procedures for Low-Concentration Samples

I. Purpose and Scope

The purpose of this guideline is to describe the packaging and shipping of low-concentration samples of various media to a laboratory for analysis.

II. Scope

The guideline only discusses the packaging and shipping of samples that are anticipated to have low concentrations of chemical constituents. Whether or not samples should be classified as low-concentration or otherwise will depend upon the site history, observation of the samples in the field, odor, and photoionization-detector readings.

If the site is known to have produced high-concentration samples in the past or the sampler suspects that high concentrations of contaminants might be present in the samples, then the sampler should conservatively assume that the samples cannot be classified as low-concentration. Samples that are anticipated to have medium to high concentrations of constituents should be packaged and shipped accordingly.

If warranted, procedures for dangerous-goods shipping may be implemented. Dangerous goods and hazardous materials pose an unreasonable risk to health, safety, or property during transportation without special handling. As a result only employees who are trained under CH2M Dangerous Goods Shipping course may ship or transport dangerous goods. Employees should utilize the HAZMAT ShipRight tool on the Virtual Office and/or contact a designated CH2M HazMat advisor with questions.

III. Equipment and Materials

- Coolers
- Clear tape
- "This Side Up" labels
- "Fragile" labels
- Vermiculite
- Ziplock bags or bubble wrap
- Ice
- Chain-of-Custody form (completed)
- Custody seals

IV. Procedures and Guidelines

Low-Concentration Samples

- A. Prepare coolers for shipment:
 - Tape drains shut.
 - Affix "This Side Up" labels on all four sides and "Fragile" labels on at least two sides of each cooler.
 - Place mailing label with laboratory address on top of coolers.
 - Fill bottom of coolers with about 3 inches of vermiculite or absorbent pads.
- B. Arrange decontaminated sample containers in groups by sample number. Consolidate VOC samples into one cooler to minimize the need for trip blanks.
- C. Affix appropriate adhesive sample labels to each container. Protect with clear label protection tape.
- D. Seal each sample bottle within a separate ziplock plastic bag or bubble wrap, if available. Tape the bag around bottle. Sample label should be visible through the bag.
- E. Arrange sample bottles in coolers so that they do not touch.
- F. If ice is required to preserve the samples, cubes should be repackaged in zip-lock bags and placed on and around the containers.
- G. Fill remaining spaces with vermiculite or absorbent pads.
- H. Complete and sign chain-of-custody form (or obtain signature) and indicate the time and date it was relinquished to Federal Express or the courier.
- J. Close lid and latch.
- K. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape.
- L. Tape cooler shut on both ends, making several complete revolutions with strapping tape. Cover custody seals with tape to avoid seals being able to be peeled from the cooler.
- M. Relinquish to Federal Express or to a courier arranged with the laboratory. Place airbill receipt inside the mailing envelope and send to the sample documentation coordinator along with the other documentation.

Medium- and High-Concentration Samples:

Medium- and high-concentration samples are packaged using the same techniques used to package low-concentration samples, with potential additional restrictions. If applicable, the sample handler must refer to instructions associated with the shipping of dangerous goods for the necessary procedures for shipping by Federal Express or other overnight carrier. If warranted, procedures for dangerous-goods shipping may be implemented. Dangerous goods and hazardous materials pose an unreasonable risk to health, safety, or property during transportation without special handling. As a result only employees who are trained under CH2M Dangerous Goods Shipping course may ship or transport dangerous goods. Employees should utilize the HAZMAT ShipRight tool on the Virtual Office and/or contact a designated CH2M HazMat advisor with questions.

V. Attachments

None.

VI. Key Checks and Items

- Be sure laboratory address is correct on the mailing label
- Pack sample bottles carefully, with adequate vermiculite or other packaging and without allowing bottles to touch
- Be sure there is adequate ice
- Include chain-of-custody form
- Include custody seals

Attachment 3

Laboratory SOPs

16-Aug-2018

SOP Effective 11/15/93
Revision 7 Effective September 2016

Verifying the Maintenance of Sample Integrity

GL-LB-E-012 Rev 7
Page 1 of 5

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

VERIFYING THE MAINTENANCE



OF

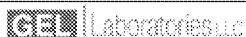
SAMPLE INTEGRITY

(GL-LB-E-012 REVISION 7)

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16-Aug-2018

Verifying the Maintenance of Sample Integrity

SOP Effective 11/15/93

Revision 7 Effective September 2016

GL-LB-E-012 Rev 7

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1.0 STANDARD OPERATING PROCEDURE FOR VERIFYING THE MAINTENANCE OF SAMPLE INTEGRITY**2.0 PURPOSE**

This document describes the procedures used to verify the effectiveness of laboratory procedures for maintaining sample integrity.

3.0 DISCUSSION

The validity of analytical results depends first on the aliquot of the sample taken for analysis being representative of the medium sampled. Assuming that the sample was collected properly, it must then be handled and stored in a manner that minimizes physical and chemical changes prior to analysis. Integrity is maintained when it can be verified that no changes have occurred to the sample.

Sample integrity must be maintained during collection, preservation, and transportation to the laboratory, and continue while the sample is being stored, handled, and analyzed in the laboratory. Sample integrity in the laboratory is maintained by a comprehensive system that integrates personnel training, building design, analytical procedures, and computer tracking procedures.

This SOP describes the computerized container tracking system, which utilizes bar codes to provide verification that the integrity of the sample is maintained while in the possession of the laboratory.

4.0 DEFINITIONS

All terminology used in this SOP is commonly used in the analytical laboratory. No special definitions are required.

5.0 PROCEDURES**5.1 Sample Login**

5.1.1 Samples are received and stored under the instructions specified in the SOP Sample Receipt, Login and Storage (GL-SR-E-001).

5.2 Sample Storage

5.2.1 The "Container Tracking" option of AlphaLIMS shall be utilized each time a sample is placed or returned to storage. This information tracks the movement of the sample container as it changes location & custody.

5.3 Sample Analysis

5.3.1 Laboratory personnel will locate and move samples from their storage locations to facilitate analysis. "Container Tracking" should be used to scan samples every time they are moved to a different location.

5.3.2 Using the "Container Tracking" option of AlphaLIMS, laboratory personnel shall scan the barcode on each bottle to positively associate each bottle with a location and an analytical batch. Even when samples are not required immediately, they must be scanned to document its location. Typing in sample IDs should be avoided, with scanning of barcoded preferred.



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5.3.3 If an analyst needs to take an aliquot out of a bottle that is in the custody of another analyst, the following procedure shall be followed.

5.3.3.1 Using the "Container Tracking" option of AlphaLIMS, the requesting analyst enters the new location and scans the desired container.

This step records the date and time of the transfer, and the name of the requesting analyst. Unless the original analyst needs the container again, the requesting analyst is now responsible for scanning and returning the sample container to the return shelf. If a container is returned to the initial analyst, it should be scanned to the returned location.

5.3.4 Sample containers shall be scanned and returned to Sample Management by the requesting analyst as soon as possible after the necessary aliquots have been taken.

5.3.5 Sample Management shall scan and return sample containers to storage as soon as possible.

5.3.6 Empty Sample Containers are scanned and returned to the empty return shelf in sample management. They are to be scanned to "Consumed by analysis/laboratory" using the "Container Tracking" option of AlphaLIMS by a member of the sample management group.

5.4 Sample Archive

5.4.1 The Sample Management Group determines which sample containers shall be transferred from sample storage to sample archive.

5.4.2 Using the "Container Tracking" option of AlphaLIMS, the Sample Custodian shall select the "Archive" location, and scan each container removed from storage.

5.5 Sample Disposal

5.5.1 The Waste Management Group determines which sample containers shall be scanned for disposal.

5.5.2 Using the "Scan To Waste Drum" option of AlphaLIMS, the waste custodian shall enter the appropriately assigned waste stream ID number (Drum Number) for that waste stream, and scan each container for disposal to be checked and verified for disposal, by LIMS. Upon LIMS approval verification, the sample will be staged for disposal.

5.5.3 The samples not approved for disposal, are segregated, and (re-)archived in accordance to section 5.4.2 of this SOP.

6.0 LABORATORY WASTE HANDLING AND DISPOSAL

Laboratory waste is disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.



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Verifying the Maintenance of Sample Integrity

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GL-LB-E-012 Rev 7

Revision 7 Effective September 2016

Page 5 of 5

7.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

No safety, health and environmental hazards are associated with the proper conduct of this activity. Refer to Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

8.0 RECORDS MANAGEMENT

Computer records are automatically recorded in duplicate and the database is backed-up nightly. Archived records are written in duplicate to tape or CD and then are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

9.0 REFERENCES

None.

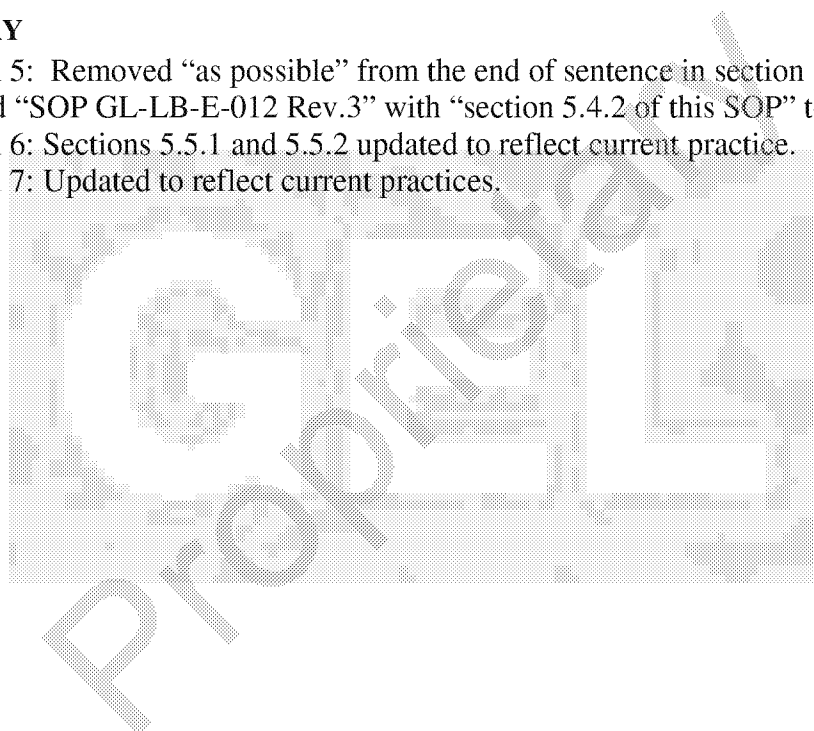
10.0 HISTORY

Revision 5: Removed “as possible” from the end of sentence in section 5.3.4.

Replaced “SOP GL-LB-E-012 Rev.3” with “section 5.4.2 of this SOP” to section 5.5.3.

Revision 6: Sections 5.5.1 and 5.5.2 updated to reflect current practice.

Revision 7: Updated to reflect current practices.



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16-Aug-2018

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE
FOR
THE DETERMINATION OF STRONTIUM 89/90
IN WATER, SOIL, MILK, FILTERS,
VEGETATION AND TISSUES
(GL-RAD-A-004 REVISION 18)

APPLICABLE TO METHODS:
EPA 600/4-80-032 Method 905.0 (Modified)
DOE RP501 Revision 1 (Modified)
EML HASL-300 (Modified)

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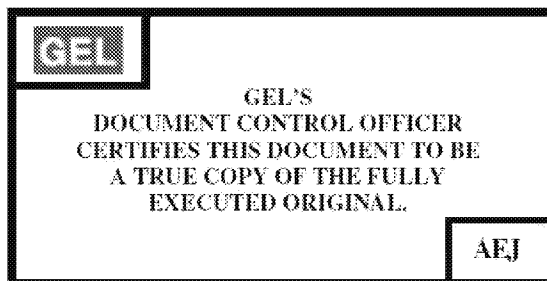


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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF STRONTIUM 89/90 IN WATER, SOIL, MILK, FILTERS, VEGETATION, AND TISSUES

2.0 METHOD OBJECTIVE, PURPOSE, AND SUMMARY

- 2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic Sr-89 and Sr-90 in water, soil, filters, vegetation, tissues, and milk.
- 2.2 This method has been modified from the source method EPA 600/4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," August 1980, Method 905.0, and uses the same principles of final source preparation, radiochemical concentration and counting. This method is also based on EML HASL-300, and is very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management samples, RP501, 1997 Edition, Revision 1.
- 2.3 This method has been modified on the basis of GEL's Performance Based Measurement System (PBMS).

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

- 3.1 Method Detection Limit (MDL): Typical minimum detectable activity (MDA) for samples analyzed for Sr-89 and Sr-90 is 2 pCi/L or 2 pCi/g.
- 3.2 Analyst training records are maintained as quality records (refer to GL-QS-E-008). Analyst training and proficiency in the method is outlined in GL-QS-E-011 for Method Validation and Initial and Continuing Demonstrations of Capability.

4.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Team Leader. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

- 5.1 Batch: Environmental samples are prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.
- 5.2 Deionized (DI) water: Type I Reagent water (Refer to GL-LB-E-016)
- 5.3 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.
- 5.4 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
- 5.5 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.

- 5.6 Matrix Spike Duplicate (MSD): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 5.7 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.
- 5.8 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.
- 5.9 Refer to SOP GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

6.0 INTERFERENCES

- 6.1 Breakthrough of strontium can occur after 45 free column volumes (FCVs)(1 FCV = 1.3 mL/column) pass through the column. To prevent breakthrough, the total volume (sum of sample loading plus rinses) passed through the column should be kept below 80% of the breakthrough volume.
- 6.2 Plutonium has been observed to retain up to 85% of the Sr Resin column under high acid conditions, which creates interferences in beta counting. This interference can be removed by passing the solution through a TRU Resin column at 2 to 6 M nitric acid. Plutonium is absorbed on the TRU Resin column; the strontium fraction elutes through the TRU Resin. The elution is then evaporated to dryness, re-dissolved in 8 M nitric acid, and passed through a Sr Resin column.
- 6.3 An excess of potassium-40 may also cause interference in subsequent beta counting due to retention of potassium on the Sr Resin column. This interference may be removed by the rinsing of the carbonate precipitate, as well as an additional 8 M nitric acid rinse on the Sr Resin column.
- 6.4 Stable strontium in the sample will compete for sites on the Sr Resin column and will affect chemical yields.
- 6.5 Ammonium ion interferes with milk. Sodium hydroxide is used instead of ammonium hydroxide to eliminate this interference.
- 6.6 Sr Resin, with 8 M nitric acid load and rinse solutions, is used to effectively remove barium-140 and potassium-40 isotopes as well as other matrix interferences. Tetravalent plutonium, neptunium, cerium, and ruthenium are not removed using nitric acid. These isotopes are effectively removed by including a rinse of 3 M nitric acid/0.05 M oxalic acid.
- 6.7 Milk fats tend to coat resin beads and clog ion exchange columns, so a batch process is used. Stable strontium is added to equilibrate the radiostrontium with the stable strontium carrier. Strontium is stripped from the resin with 8 M nitric



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acid, converted to carbonate to concentrate the strontium, and separated from calcium and other elements by Sr Resin.

7.0 SAFETY PRECAUTIONS AND WARNINGS

- 7.1 Personnel performing this analytical procedure are trained to the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.
- 7.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.
- 7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.
- 7.4 If there is any question regarding the safety of any laboratory practice, stop immediately, and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

- 8.1 Apparatus and Equipment
 - 8.1.1 Stainless steel planchets (1" x 3/32")
 - 8.1.2 Glass fiber filters
 - 8.1.3 Hot plates
 - 8.1.4 Centrifuge tubes (plastic)
 - 8.1.5 Beakers (glass and Teflon of varying sizes)
 - 8.1.6 Large glass chromatography columns
 - 8.1.7 Drying lamps/drying oven
 - 8.1.8 0.45 μ m 25 mm diameter pre-weighed filter in disposable filter funnel
 - 8.1.9 47 mm filter funnels
 - 8.1.10 Sr Resin® columns EiChrom Industries Inc. or o2si equivalent
 - 8.1.11 Teflon-coated stirring magnets
 - 8.1.12 Petri dishes
 - 8.1.13 2.5 cm³ column
 - 8.1.14 25 mL column funnel extension
 - 8.1.15 Filtration apparatus
- 8.2 Instrumentation
 - 8.2.1 Gas-flow proportional counting system
 - 8.2.2 Analytical balance

9.0 REAGENTS AND STANDARDS

- 9.1 Reagents



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- 9.1.1 Anion exchange resin, Analytical Grade 1X8, chloride form, 50-100 mesh and 100-200 mesh.
- 9.1.2 Ammonium hydroxide (NH₄OH), concentrated
- 9.1.3 50% Ammonium hydroxide solution: Slowly add 500 mL concentrated ammonium hydroxide to 500 mL DI water.
- 9.1.4 Calcium nitrate (1.25 M), calcium carrier: Dissolve 295 g of calcium nitrate tetrahydrate [Ca(NO₃)₂•4H₂O] in 500 mL DI water and dilute to 1 L with DI water.
- 9.1.5 Cation resin, Marthon C, sodium form, or equivalent. Use 20-50 mesh material for easier recovery from the milk.
- 9.1.6 Deionized (DI) water
- 9.1.7 pH 10 DI water: Slowly adjust pH with 10 M sodium hydroxide.
- 9.1.8 80% Ethanol: Dilute 800 mL EtOH to 1 L with DI water.
- 9.1.9 Hydrochloric acid (HCl), concentrated, 12 M
- 9.1.10 Hydrochloric acid (HCl) 9 M: Add 750 mL concentrated HCl (12 M) to 250 mL of DI water.
- 9.1.11 Hydrofluoric acid (HF), concentrated
- 9.1.12 Hydrogen peroxide (30% H₂O₂)
- 9.1.13 Nitric acid (HNO₃), concentrated, 16 M
- 9.1.14 Nitric acid, 8 M: Add 500 mL concentrated nitric acid (16 M HNO₃) to 400 mL DI water, allow to cool, and dilute to 1000 mL with DI water.
- 9.1.15 3 M Nitric acid/0.05 M Oxalic acid solution: Carefully add 188 mL of concentrated HNO₃ (16 M) and add 6.3 g of oxalic acid dihydrate to 800 mL of DI water and dilute to 1 L with DI water.
- 9.1.16 Nitric acid, 2 M: Add 125 mL concentrated nitric acid (16 M HNO₃) to 500 mL DI water and dilute to 1000 mL with DI water.
- 9.1.17 Nitric acid, 0.05 M: Carefully mix 3.1 mL 16 M HNO₃ (concentrated) with 500 mL DI water and dilute to 1000 mL with DI water.
- 9.1.18 Methyl orange pH indicator
- 9.1.19 1 M Oxalic acid: Dissolve 126.07 g oxalic acid dihydrate in 500 mL DI water and dilute to 1000 mL.
- 9.1.20 Phenolphthalein solution
- 9.1.21 Sodium carbonate (Na₂CO₃), 0.75 M: Add 79.5 g Na₂CO₃ to 1000 mL with DI water.
- 9.1.22 Sodium hydroxide (NaOH), 6 M: Cautiously add 120 g NaOH pellets to approximately 300 mL water. When cool, dilute to 500 mL.



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- 9.1.23 Sodium hydroxide (NaOH), 10 M. Add 400 g of NaOH pellets to 800 mL of DI water and mix. Dilute to 1 L when cool.
- 9.1.24 Strontium carrier (standardized), 10 mg/mL: Dissolve 24.16 g $\text{Sr}(\text{NO}_3)_2$ in DI water and dilute to 1000 mL. Alternatively use 10 Sr mg/mL o2si standard.
- 9.1.25 Thymol blue pH indicator
- 9.1.26 Yttrium carrier (standardized), 9 mg/mL: Add 11.43 g yttrium oxide (Y_2O_3) to a new beaker containing 20 mL DI water. Heat to boiling on a magnetic stirring hot plate while adding concentrated nitric acid in small amounts. Approximately 20 mL of concentrated nitric acid will be necessary to dissolve the yttrium oxide. Small additions of DI water may be required to replace what was lost by evaporation. After total dissolution, add 30 mL concentrated nitric acid and dilute to 1 L with DI water. Alternatively, use 10 Y mg/mL o2si standard.

9.2 Standards

Refer to GL-RAD-M-001 for instructions concerning the preparation of standard solutions.

9.2.1 NIST traceable Strontium-89 standard

9.2.2 NIST traceable Strontium-90 standard

10.0 SAMPLE HANDLING AND PRESERVATION

- 10.1 Water samples should be collected in plastic bottles and preserved with concentrated nitric acid to pH < 2.
- 10.2 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing the sample on a pH strip. **DO NOT** insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should contact the Group Leader or Team Leader. If approved by the client, the analyst should adjust the pH with nitric acid to a pH < 2. If the sample is pH adjusted let the sample sit in the original container for a minimum of 24 hours before analysis.
- 10.3 Milk samples should be preserved with sodium bisulfate at approximately 40 grams/gallon, so that storage in a refrigerator is not necessary. Samples that are not preserved at collection must be kept refrigerated until preserved.

11.0 SAMPLE PREPARATION

NOTE: Sample aliquot size may be estimated using the count time estimator spread sheet.

NOTE: Spiking and tracing steps should be witnessed by either another analyst qualified in this procedure or the Team Leader/Group Leader responsible for this procedure. After



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adding tracers and spikes, the witness must initial and record the date and time of witnessing.

11.1 Sample preparation techniques for water matrix.

11.1.1 Transfer an appropriate aliquot of water sample to a glass beaker and record the volume. If required, the DUP, and MS and MSD should be the same aliquot as the appropriate sample referenced. Prepare a MB and LCS by using DI water and concentrated nitric acid to a pH < 2. The MB and LCS volumes should be equivalent to the largest aliquot in the batch and should be recorded.

11.1.2 Add 0.5 mL of strontium carrier to each sample including MB and LCS. Add an appropriate amount of Sr-90 and/or Sr-89 spike to MS, LCS, and LCSD as applicable. Reference batch pull sheet for client requirements to determine appropriate spikes needed for the batch.

NOTE: Due to the short half life of Sr-89, if both Sr-89 and Sr-90 are being added as spikes, the volume to be added is determined by the decay corrected value in AlphaLIMS so that the dpm values for both isotopes are similar. If only adding Sr-90, then the typical volume added is 0.1 mL.

11.1.3 Add 3 to 4 drops of phenolphthalein solution, and then add 6 M NaOH dropwise until the sample turns a light pink color. Stir. Do not add NaOH in extreme excess.

11.1.4 Add 10 mL of 0.75 M sodium carbonate and stir.

NOTE: For samples that do not normally precipitate, add 2 to 3 mL of calcium carrier to all samples.

11.1.5 Cover the beaker with a watch glass and heat to rapid boiling.

11.1.6 Remove the watch glass and allow the precipitate to settle and cool for at least one hour, or overnight, if possible.

11.1.7 Aspirate the supernate then transfer the precipitate into a centrifuge tube using pH 10 DI water.

11.1.8 Centrifuge and discard the supernate.

11.1.9 Rinse the sides of the beaker with 15 mL 8 M nitric acid. Transfer the rinse to the centrifuge tube. This is the load solution for the Sr Resin column.

11.1.10 Proceed to Step 11.7, Strontium Determination.

11.2 Sample preparation techniques for soil matrix.

11.2.1 Leach method

11.2.1.1 Aliquot dried and homogenized sample into glass beaker. Record aliquot information. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample



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referenced. Record the aliquot for the MB and LCS as the largest aliquot in the batch.

- 11.2.1.2 Add 0.5 mL of strontium carrier to each sample including MB and LCS. Add an appropriate amount of Sr-90 and/or Sr-89 spike to MS, MSD, LCS and LCSD as applicable. Reference batch pull sheet for client requirements to determine appropriate spike.

NOTE: Due to the short half life of Sr-89, if both Sr-89 and Sr-90 are being added as spikes, the volume to be added is determined by the decay corrected value in AlphaLIMS so that the dpm values for both isotopes are similar. If only adding Sr-90, then the typical volume added is 0.1 mL.

- 11.2.1.3 For sample aliquots > 0.5 g, it is recommended that the samples be ashed in a muffle furnace as specified in GL-RAD-A-021B.

- 11.2.1.4 Slowly add 20 to 50 mL of 8 M nitric acid to the beaker. Cover the sample with a watch glass and reflux, for approximately 30 minutes.

NOTE: When leaching concrete samples, concentrated nitric acid should be used instead of 8 M nitric acid due to the matrix of the sample.

- 11.2.1.5 Slowly add 2 to 3 drops of 30% hydrogen peroxide, swirl, and continue refluxing for approximately 30 minutes.
- 11.2.1.6 Repeat step 11.2.1.5 at least two more times.
- 11.2.1.7 Slurry the sample and transfer the mixture to a centrifuge tube.
- 11.2.1.8 Centrifuge the mixture to separate the leachate from the remaining soil. Transfer the leachate to a glass beaker.
- 11.2.1.9 Add 10 mL 8 M nitric acid to the centrifuge tube, cap and mix. Repeat Step 11.2.1.8.
- 11.2.1.10 Discard any remaining solid matter.
- 11.2.1.11 Heat the leached sample in the glass beaker to dryness on a hot plate.
- 11.2.1.12 Dissolve the residue in the glass beaker with 10 to 15 mL of 8 M nitric acid. Cover and reflux for approximately 15 minutes.
- 11.2.1.13 Remove samples from the hot plate and allow to cool. This is the load solution for the strontium column.
- 11.2.1.14 Proceed to Step 11.7, Strontium Determination



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11.2.2 Preparation of large soil aliquots

- 11.2.2.1 Perform Steps 11.2.1.1 through 11.2.1.11 above.
- 11.2.2.2 Dissolve the sample residue from Step 11.2.1.11 in approximately 5 mLs of concentrated hydrochloric acid. Heat to dryness. Dissolve the sample residue in 10 to 15 mL of 9 M hydrochloric acid. This is the load solution for the anion exchange column.
- 11.2.2.3 Prepare the anion exchange column (large 7 cm columns) by filling the column to the top with anion exchange resin. Rinse the column with approximately 25 mL of 9 M hydrochloric acid.
- 11.2.2.4 Place a clean, labeled centrifuge tube under the anion column. Pass the sample through the anion column collecting it in the centrifuge tube.
- 11.2.2.5 Rinse the column with 15 to 20 mL of 9 M hydrochloric acid, and collect it in the centrifuge tube.
- 11.2.2.6 Take the volume in the centrifuge tube to dryness in a beaker on a hot plate.
- 11.2.2.7 Dissolve the residue in approximately 5 mL of concentrated nitric acid and evaporate to dryness. Dissolve the residue in the glass beaker with 10 to 15 mL of 8 M nitric acid. Cover and reflux for approximately 15 minutes.
- 11.2.2.8 Remove samples from the hot plate and allow to cool. This is the load solution for the strontium column.
- 11.2.2.9 Proceed to Step 11.7, Strontium Determination.

11.3 Sample preparation techniques for milk matrix.

- 11.3.1 Transfer appropriate aliquot of milk sample to a glass beaker and record volume. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Prepare a MB and LCS by using DI water and concentrated nitric acid to a pH < 2. The MB and LCS volumes should be recorded.
- 11.3.2 Add 1.0 mL of strontium carrier to each sample including the MB and LCS. Add an appropriate amount of Sr-90 and/or Sr-89 spike to MS, MSD, LCS as applicable. Reference batch pull sheet for client requirements to determine appropriate spike.

NOTE: Due to the short half life of Sr-89, if both Sr-89 and Sr-90 are being added as spikes, the volume to be added is determined by the decay corrected

value in AlphaLIMS so that the dpm values for both isotopes are similar. If only adding Sr-90, then the typical volume added is 0.1 mL.

- 11.3.3 Add a Teflon-coated stirring magnet and stir for approximately 15 minutes.
- 11.3.4 Add 30 grams of Cation Exchange Resin Na⁺ form, spherical beads (20-50 mesh), and continue stirring for at least one hour. Stirring must be vigorous enough to distribute the resin throughout the sample.
- 11.3.5 Remove the beaker from the stirrer and allow the resin to settle.
- 11.3.6 Slowly decant and discard the milk, ensuring that no resin is lost. Rinse the beaker with several (3 to 5) 600 mL portions of hot water, continuing until the rinse is clear. It is necessary that the milk be completely removed from the resin.
- 11.3.7 Transfer the resin to a large chromatography column that has been fitted with a control valve on the bottom. Allow the excess rinse water in the resin drain out. Discard the excess rinse water.
- 11.3.8 Place 1 liter beakers under columns. Elute the strontium from the resin by passing two 200 mL volumes of 8 M nitric acid through the column at a flow rate of 1 to 2 drops per second. Collect the effluent.

NOTE: If the flow rate exceeds 1 to 2 drops per second, the strontium will not be completely removed from the resin. Slower flow rates will not adversely affect the elution.

- 11.3.9 Evaporate the effluent to a volume of approximately 50 mL. Some insoluble material may be present. Dilute to at least 500 mL with DI water.
 - 11.3.10 Add 3 to 4 drops of phenolphthalein solution, and then add 10 M sodium hydroxide until the sample turns a light pink color, stir.
 - 11.3.11 Slowly add 50 mL of 0.75 M sodium carbonate while stirring.
 - 11.3.12 Cover the beaker with a watch glass and heat to rapid boiling.
 - 11.3.13 Remove the watch glass and allow the sample to cool for at least one hour, or overnight if possible, to allow all insoluble material to settle out.
 - 11.3.14 Aspirate the supernate, and then transfer the voluminous precipitate into a centrifuge tube and centrifuge. Discard the supernate.
 - 11.3.15 Wash the precipitate with approximately 35 mL of pH 10 DI water, ensuring that the precipitate is thoroughly re-suspended in the wash.
- NOTE:** This rinsing ensures removal of potassium prior to loading onto Sr column.
- 11.3.16 Centrifuge and discard supernate and repeat Step 11.3.15.



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NOTE: Most milk contains 1400 to 1700 mg of potassium (K) per liter. Because the ionic radii of potassium and strontium are very close, potassium will compete with strontium for resin sites on the Sr resin. Therefore, removal of potassium is necessary to prevent extremely low strontium yields.

11.3.17 Discard supernate.

11.3.18 In small portions, very carefully add 20 to 30 mL of 8 M nitric acid until the precipitate is dissolved. Copious amounts of carbon dioxide will be evolved, and care must be taken to avoid loss of sample due to spattering.

11.3.19 Proceed to Step 11.7, Strontium Determination.

11.4 Preparation technique for air filters

11.4.1 Filters should be prepared in accordance with GL-RAD-A-026. Transfer an appropriate aliquot of the digested air filter to a glass beaker and record the volume. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Prepare a MB and LCS by using DI water and concentrated nitric acid to a pH < 2. The MB and LCS volumes should be equivalent to the largest aliquot in the batch and should be recorded.

NOTE: If filter is not completely digested, consult TL or GL for additional instructions.

11.4.2 Add 0.5 mL of strontium carrier to each sample including MB and LCS. Add an appropriate amount of Sr-90 and/or Sr-89 spike to MS, MSD, LCS and LCSD as applicable. Reference batch pull sheet for client requirements to determine appropriate spike.

NOTE: Due to the short half life of Sr-89, if both Sr-89 and Sr-90 are being added as spikes, the volume to be added is determined by the decay corrected value in AlphaLIMS so that the dpm values for both isotopes are similar. If only adding Sr-90, then the typical volume added is 0.1 mL.

11.4.3 Add 3 to 4 drops of phenolphthalein solution, and then add 6 M sodium hydroxide dropwise while stirring until the sample turns a light pink color. Do not add NaOH in extreme excess.

11.4.4 Slowly add 10 mL of 0.75 M sodium carbonate.

11.4.5 Cover the beaker with a watch glass and heat to rapid boiling.

11.4.6 Remove the watch glass and allow the precipitate to settle and cool for at least one hour, or overnight if possible.

11.4.7 Aspirate the supernate, then transfer the precipitate into a centrifuge tube using pH 10 DI water.

11.4.8 Centrifuge and discard the supernate.



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- 11.4.9 Rinse the sides of the beaker with 10-15 mL 8 M nitric acid. Transfer the rinse to the centrifuge tube. This is the load solution for the Sr Resin column.
- 11.4.10 Proceed to Step 11.7, Strontium Determination.
- 11.5 Preparation technique for tissue samples and vegetation.
- 11.5.1 Aliquot dried and homogenized sample into glass beaker for ashing. Record aliquot information. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Record the aliquot for the MB and LCS as the largest aliquot in the batch.
- 11.5.2 Add 0.5 mL of strontium carrier to each sample including MB and LCS. Add an appropriate amount of Sr-90 and/or Sr-89 spike to MS, MSD, LCS and LCSD as applicable. Reference batch pull sheet for client requirements to determine appropriate spike.
- NOTE:** Due to the short half life of Sr-89, if both Sr-89 and Sr-90 are being added as spikes, the volume to be added is determined by the decay corrected value in AlphaLIMS so that the dpm values for both isotopes are similar. If only adding Sr-90, then the typical volume added is 0.1 mL.
- 11.5.3 Ash samples in accordance with GL-RAD-A-021B.
- NOTE:** Ashing of tissue and vegetation can take significantly longer than normal soils and the majority of sample should be gray or white when complete.
- 11.5.4 Once ashing is complete, add 300 mL of 8 M nitric acid and 1 mL of 1.25 M calcium nitrate.
- 11.5.5 Cover the sample and reflux for at least 1 to 2 hours. Allow to cool and filter through a glass fiber filter. Collect the filtrate.
- 11.5.6 Boil the filtrate and evaporate to approximately 25 to 50 mL. Dilute to approximately 700 mL with DI water.
- 11.5.7 Add 1 mL of methyl orange and 10 g of oxalic acid.
- NOTE:** For the above step, the 10 g of oxalic acid can be dissolved in 50 mL DI water and the dissolved solution added to the sample. Larger volumes of solution can be made in accordance to batch size.
- 11.5.8 Add ammonium hydroxide until the solution turns yellow, giving a pH of approximately 4. Allow the samples to sit overnight.
- 11.5.9 Aspirate the supernate and transfer the precipitate to a centrifuge tube. Centrifuge the sample and discard the supernate.
- 11.5.10 Rinse the precipitate with DI water and centrifuge. Discard supernate.
- 11.5.11 Dissolve the precipitate in 50 mL of 8 M nitric acid. Transfer to a 250 mL beaker and evaporate to dryness very slowly.

- 11.5.12 Wet ash the sample with 5 to 10 mL of concentrated nitric acid and 1 mL of hydrogen peroxide. (Repeat this step until the sample no longer produces a brownish gas.)
- 11.5.13 Evaporate to dryness.
- 11.5.14 Ash the samples in a furnace at approximately 500° C for at least 30 minutes. Allow the samples to cool.
- 11.5.15 Dissolve the sample residue with 15 mL of 8 M nitric acid and reflux on a hot plate until the solids dissolve. This is the load solution for the strontium column.
- 11.5.16 Proceed to Step 11.7, Strontium Determination.
- 11.6 Preparation technique for samples previously run and reported extremely high alpha Pu⁺, Np⁺, Ce⁺:
 - 11.6.1 Pour aliquot. Spike appropriate samples.
 - 11.6.2 Add 0.5 mL Sr carrier, 1 mL iron carrier, 5 mL concentrated nitric acid.
 - 11.6.3 Reflux 1 hour. Add concentrated ammonium hydroxide in excess until iron precipitates. Centrifuge out the iron and keep the supernate.
 - 11.6.4 Evaporate the sample to dryness and dissolve residue in approximately 10 mL of 2 M nitric acid. This is the load solution for Tru-spec columns.
 - 11.6.5 Prepare Tru-Spec columns by rinsing with 10 mL of 2 M nitric acid.
 - 11.6.6 Pour load solution through column and collect in centrifuge tube.
 - 11.6.7 Pour sample back into beakers and evaporate to dryness. Dissolve residue in approximately 10 mL of 8 M nitric acid. This is the load solution for Sr columns. Proceed to step 11.7, Strontium Determination.
- 11.7 Strontium Determination
 - 11.7.1 For each sample, prepare a 25 mm pre-weighed filter with funnel. Label each funnel and record the weight.

NOTE: For milk samples, large vegetation and tissue samples, use Sr columns with double the amount of Sr resin. Additionally, all rinse and eluent volumes must be doubled.

 - 11.7.2 Condition the Sr Resin column by rinsing it with 5 mL of 8 M nitric acid.
 - 11.7.3 When the rinse has passed completely through the column, load the sample onto the column.
 - 11.7.4 When the sample has passed completely through the column, rinse the tube with 5 to 10 mL of 8 M nitric acid, depending on sample color. (An additional 10 mL 8 M nitric acid rinse may be used to remove excess iron and potassium from the column.)

- 11.7.5 When the rinse has passed completely through the column, rinse the column with 10 mL of 3 M nitric acid/0.05 M oxalic acid. Discard the rinse.
- 11.7.6 When the rinse has passed completely through the column, rinse the column with 5 mL 8 M nitric acid.
- NOTE:** This additional 8 M rinse removes any residual oxalic acid and ensures full removal of K^+ and Ba^{+2} that may be present.
- 11.7.7 When the rinse has passed completely through the column, place a clean labeled centrifuge tube under the column and elute the strontium with 15 mL of 0.05 M nitric acid. Record the date and time for the beginning of Y-90 in-growth as Sr separation. Save the labeled Sr Resin column for later Y-90 separation, if necessary.
- 11.7.7.1 If Sr-89 is required, add DI water to the column for preservation of the resin, then cap and save.
- NOTE:** The remaining steps should be performed as quickly as possible to avoid excess Y-90 growth. Y-90 in-grows at the rate of 1% an hour.
- 11.7.8 Add 2 drops of thymol blue indicator then add 6 M sodium hydroxide dropwise until the samples turn blue. Add 3 mL 0.75 M sodium carbonate and heat in the microwave until warm. Allow precipitate to settle for approximately 30 minutes.
- 11.7.8.1 After the strontium has been precipitated coming off of the Sr Resin column, if there is visibly too much precipitate, these steps can be taken:
- 11.7.8.1.1 Centrifuge the sample and decant the supernate.
- 11.7.8.1.2 Dissolve the precipitate in 15 to 20 mL concentrated nitric acid and allow to sit in a refrigerator overnight.
- 11.7.8.1.3 The next day centrifuge the sample and discard the supernate. (Should have strontium nitrate, $SrNO_3$.) Dissolve the precipitate in 8 M nitric acid and rerun through a Sr Resin column using the standard procedure.
- 11.7.9 Filter the samples through the pre-weighed filter rinsing the centrifuge tube with pH 10 DI water. Rinse the filter funnel with a minimum amount of pH 10 water. Rinse the filter funnel with minimum amount of 80% ethanol.
- 11.7.10 Allow filters to air dry in petri dishes. Weigh each filter and record the weight. Place the filter in a steel planchette and into the labeled petri

dish. Calculate the net weight on the filter to determine the strontium yield.

- 11.7.11 Turn filters and completed batch paperwork to count room for Gas Flow Proportional counting analysis.

11.8 Y-90 Isolation After In-growth

NOTE: If Sr-89 is known to be absent, or if total strontium is requested, the final result can be determined with a single count. However, if Sr-89 is present, Sr-90 is measured by isolating Y-90 using this section and counting the Y-90.

- 11.8.1 After total strontium has been determined, store the filters for a minimum of 5 to 7 days, to allow for Y-90 in-growth.
- 11.8.2 Remove the filter from the planchette and place in a labeled centrifuge tube with 10 mL of 8 M nitric acid. Shake well and allow the precipitate to completely dissolve. Remove the filter from the centrifuge tube using forceps. Rinse the filter into the centrifuge tube with approximately 5 mL of 8 M nitric acid. Condition column by adding 0.5 mL of yttrium carrier to each centrifuge tube and swirl.
- 11.8.3 For each sample, prepare a 25 mm pre-weighed filter with funnel and record the weight.
- 11.8.4 Recondition the same column used for the initial strontium separation by adding 5 mL of DI water into each column. Allow to drain. Condition column by adding 5 mL of 8 M nitric acid and allow to drain.
- NOTE:** The DI water removes Bi-210 in-growth from any Pb-210 that may be tightly bound to the resin.
- 11.8.5 Place a clean labeled centrifuge tube under each column. Load the sample into the column, rinse the centrifuge tube with 1 to 2 mL of 8 M nitric acid. Record the date and time as Y separation. This marks the end of the Y-90 in-growth and the start of the Y-90 decay.
- 11.8.6 When the sample has passed through the column, precipitate the yttrium by adding 1 drop of thymol blue indicator and 1 mL of 1 M oxalic acid. Add 2 mL concentrated ammonium hydroxide and swirl. Continue to precipitate samples by adding 50% ammonium hydroxide drop wise until samples reach a pH of 2.7. (This is indicated by a color change from pink to light orange, and checked with narrow range pH strips.) Allow the precipitate to settle for approximately 30 minutes. (Do not add ammonium hydroxide in excess.)
- 11.8.7 Swirl the centrifuge tube to mix the precipitate. Filter the sample through the pre-weighed filter, rinse the centrifuge tube and sides of the funnel with DI water. Rinse the filter funnel with 80% ethanol.



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11.8.8 Allow filters to air dry in petri dish. Weigh each filter and record the weight. Place filter in steel planchette and into the labeled petri dish. Calculate the net weight on the filter to determine the yttrium yield.

11.8.9 Turn in samples and completed batch paperwork to the count room for gas flow proportional analysis.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

NOTE: Client contractual QC requirements override the requirements in this section.

12.1 Analyst and Method Verification Requirements

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Control Requirements

12.2.1 A Method Blank (MB) should accompany each batch of 20 or fewer samples. The reported value of the blank should be less than or equal to the Contract Required Detection Limit (CRDL).

12.2.2 The carrier added to all samples is used to calculate the chemical yield. The chemical yield of all samples should be between 25-125%.

12.2.3 A Duplicate (DUP) sample should be run with each batch of 20 or less samples. The Relative Percent Difference (RPD) between the actual sample and the DUP should be less than or equal to 20% if both the sample and the DUP results are greater than 5 times MDC or 100% if they are both less than 5 times MDC. If both results are less than MDC then limits on RPD are not applicable.

12.2.4 A Laboratory Control Sample (LCS) should be run with each batch of 20 or less samples. The recovery of the LCS should fall between 75-125%.

12.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the QC criteria cannot be satisfied, the analyst should inform the Group Leader and initiate a Data Exception Report as outlined in GL-QS-E-004.

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE

13.1 Refer to appropriate counting procedure GL-RAD-I-006 or GL-RAD-I-016 for instrument calibration and performance.

13.2 Standardization of Sr carrier.

13.2.1 Pipet four aliquots (typically 1.0 mL) of strontium carrier (nom. conc. 10 mg Sr/mL) into tared 50 mL centrifuge tubes. Record the weight of each carrier aliquot.

13.2.2 Dilute to 30 mL with DI water. Add 2 drops of thymol blue indicator (or phenolphthalein). Add 6 M sodium hydroxide drop wise until the solution turns blue (or pink if using phenolphthalein). Add additional sodium hydroxide drop wise if necessary until the indicator changes



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color. Add 5 mL of 0.75 M sodium carbonate to precipitate strontium carbonate. Heat in the microwave for approximately 20 to 30 seconds. Set aside for at least 15 minutes.

13.2.3 Filter through a pre-weighed tared Gooch crucible containing a double 2.1 cm glass fiber filter. Rinse with two 5 mL portions of pH 10 DI water, and one 5 mL portion of 80% alcohol.

13.2.4 Dry in the oven for approximately 30 minutes. Cool. Weigh.

13.2.5 Calculate the standard weight of strontium carrier in mg/mL of solution to obtain the standard weight for 1.00 mL of strontium carrier solution. Acceptable precision is a standard deviation of less than 1% of mean value. Record the carrier Reference Material number and the standardization results in the appropriate spreadsheet. Label the carrier solution with the standardization results.

13.3 Standardization of Y carrier.

13.3.1 Pipet four aliquots (typically 1.0 mL) of yttrium carrier (nom. conc. 9 mg Y/mL) into tared 50 mL centrifuge tubes. Record the weight of each carrier aliquot.

13.3.2 Dilute to 30 mL with water. Add 2 drops thymol blue indicator. Add 5 mL of 1 M oxalic acid. Add ammonium hydroxide drop wise until the indicator changes from pink to light orange.

NOTE: The desired pH is 2.7, which is necessary to ensure that yttrium oxalate precipitates with the correct stoichiometric form.

13.3.3 Heat in the microwave for approximately 20 to 30 seconds. Set aside for at least 15 minutes.

13.3.4 Filter through a pre-weighed tared Gooch crucible containing a double 2.1 cm glass fiber filter. Rinse with two 5 mL portions of DI water, and one 5 mL portion of 80% alcohol.

13.3.5 Dry in the oven for approximately 30 minutes. Cool. Weigh.

13.3.6 Calculate the standard weight of yttrium carrier in mg/mL of solution to obtain the standard weight for 1.00 mL of yttrium carrier solution. Acceptable precision is a standard deviation of less than 1% of mean value. Record the carrier Reference Material number and the standardization results in the appropriate spreadsheet. Label the carrier solution with the standardization results.

13.4 Preparation of Sr-89 Standards.

NOTE: Eppendorf pipets are used to quickly add estimated amount of carrier in the preparation of standards. To insure highest accuracy, the amount added is calculated by

weight. It is important to follow the filter preparation and precipitate washing and rinsing steps carefully to ensure highest accuracy in yield determination.

- 13.4.1 Weigh and record an aliquot of Sr-89 tracer (10,000 dpm) into a disposable centrifuge tube. Add varying amounts of Sr carrier into each tube. Record the information on the standard prep sheet.
 - 13.4.2 Dilute to 30 mL with water and add 2 drops of thymol blue. Add 6 M sodium hydroxide drop wise until the sample turns blue. Do not add sodium hydroxide in excess. Then add 3 mL of 0.75 M sodium carbonate. Heat for approximately 20 to 30 seconds in the microwave, or at least 5 minutes in a hot water bath. Allow the precipitate to stand for at least 15 minutes.
 - 13.4.3 Place a pre-weighed filter with filter cup into assembly, rinse the assembly with 5 mL of 80% ethanol, and connect the vacuum. Aspirate to dryness and disconnect vacuum. Swirl the centrifuge tube containing the standard to be filtered, and pour the entire sample into the filter reservoir. Apply and aspirate to dryness.
 - 13.4.4 Disconnect vacuum. Rinse the filter with 5 mL of pH 10 DI water. Connect the vacuum and aspirate to dryness. (Repeat three times.)
 - 13.4.5 Disconnect vacuum. Rinse the filter with 5 mL of 80% ethanol. Connect vacuum and aspirate to dryness. Continue to rinse funnel assembly with 80% ethanol as necessary to recover any precipitate clinging to walls.
 - 13.4.6 With vacuum connected, remove the reservoir and rinse the edges of the filter with 80% ethanol. Disconnect vacuum and remove filter. Then allow filter to air dry.
 - 13.4.7 Weigh filter to calculate yield and submit for counting.
- 13.5 Preparation of Y-90 and Sr-90 sources for instrument calibration.

NOTE: Calibration standards should be prepared without the use of vacuum boxes. Gravity flow rates are recommended.

- 13.5.1 Add a known amount of Sr-90 standard that has Y-90 in equilibrium to a centrifuge tube that contains 15 mL of 8 M nitric acid. Use an amount that will provide at least 10,000 counts measured on the detector in a short period of time.
- 13.5.2 Prepare a predetermined amount of Sr-Spec columns (usually 8) by pre-rinsing with 5 mL of 8 M nitric acid.
- 13.5.3 After the Sr-Spec column has drained completely, place a clean centrifuge tube under the column and transfer the contents of the centrifuge tube containing the load solution to the column. Record start time for yttrium-90 decay.



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- 13.5.4 Rinse the centrifuge tubes with 2 mL of 8 M nitric acid and transfer to the Sr-Spec columns.
- 13.5.5 Add 5 mL 8 M nitric acid and allow to drain to the centrifuge tube.
- 13.5.6 Add 10 mL of 3 M nitric acid and allow to drain to the centrifuge tube.
- 13.5.7 Remove the centrifuge tube from under the columns and save for Y-90 precipitation. Place new centrifuge tubes under the columns.
- 13.5.8 Elute the Sr-90 from the column with 15 mL of 0.05 M nitric acid then rinse with a final 5 mL of 0.05 M nitric acid and combine the two elutions. Record this time as the Sr-90/Y-90 separation time.
- 13.5.9 Add varying amounts of stable strontium carrier to the strontium fractions and add 0.5 mL of stable yttrium carrier to the yttrium fractions.
- 13.5.10 Proceed to step 11.7.8 for strontium source preparation and to step 11.8.6 for yttrium source preparation. When source preparation is complete, the filters are ready for counting on the GFPC detectors.

14.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

Refer to the appropriate counting procedure GL-RAD-I-006 or GL-RAD-I-016 for instrument operation.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-010 for instrument maintenance.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation, and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Data are reviewed and packaged in accordance with GL-RAD-D-003.

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

- 20.1 B.D. Stewart, "Preparation of Milk Samples for Strontium Analysis," Sr-01, Radiochemistry Procedures Manual, Arizona State University Radiation Measurements Facility, 1992.
- 20.2 DOE Methods for Evaluating Environmental and Waste Management Samples, RP501, 1997 Edition.

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- 20.3 DOE EML Procedures Manual, HASL-300, 28th Edition, 1997.
- 20.4 Los Alamos National Laboratories Methods Manual.
- 20.5 Eichrom technical method data.
- 20.6 Special thanks to Dr. Frank Kinard with the College of Charleston for his help in reviewing the strontium method.

21.0 HISTORY

Revision 14: Updated the volume concentration of Nitric acid used in section 11.7.6.
Inserted New NOTE: If excessive ammonium hydroxide was used during actinide scavenge then additional oxalic acid will need to ensure complete color change of indicator. Sample should be pink at this time.

Revision 15: Updated sections 13.2.5 and 13.3.6 to read acceptable precision is a standard deviation of less than 1% of the mean value.

Revision 16: Removed section regarding tandem analysis of APU/Sr and updated technical details of milk analysis.

Revision 17: Technical updates for SOP consistency as part of annual review.

Revision 18: Removed reference to Queue Sheets.



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APPENDIX 1

STRONTIUM DETERMINATION

Use Sr resin column

Column Work

- _____ 5 mL 8 M HNO₃ (conditioning)
- _____ Load solution: 10 to 15 mL 8 M HNO₃
- _____ Rinse 10 mL 8M HNO₃ (Additional 10 mL 8 M HNO₃ may be used to remove excess iron and potassium from column, if necessary)
- _____ Rinse: 10 mL 3 M HNO₃/0.05 M Oxalic Acid
- _____ Rinse: 5 mL 8 M HNO₃
- _____ **Elute:** 15 mL 0.05 M HNO₃ and catch in a c-tube(Record date and time)
- _____ Add 2 drops of Thymol Blue indicator and 6 M NaOH dropwise until blue
- _____ Add 3 mL 0.75 M sodium carbonate and heat in microwave until warm
- _____ Wait approximately 30 minutes
- _____ Filter



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APPENDIX 2

YTTRIUM ISOLATION AFTER IN-GROWTH

Use saved Sr Resin column from 1st separation

Column Work

- _____ Rinse: 5 mL DI water
- _____ Rinse: 5 mL 8 M HNO₃ conditioning
- _____ Load: 8 M HNO₃ and catch in C-tube (Record date and time)
- _____ Rinse: 1 to 2 mL 8 M HNO₃ and catch in C-tube
- _____ To volume in C-tube, add 1 drop Thymol Blue and 1 mL 1 M Oxalic Acid
- _____ Add 2 mL concentrated ammonium hydroxide and swirl
- _____ Add 50% ammonium hydroxide dropwise until approximately pH 2.7
- _____ Wait approximately 30 minutes
- _____ Filter



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APPENDIX 3

ANION EXCHANGE COLUMN FOR LARGE SOIL ALIQUOTS

Use 7 cm column with 1X8 anion exchange resin (Cl⁻ form 100-200 mesh)

Column Work:

- _____ 25 mL 9 M HCl (conditioning)
- _____ Load 9 M sample solution onto column and collect in C-tube
- _____ Rinse with 15 to 20 mL 9 M HCl and collect in C-tube
- _____ Take collected volume dry
- _____ Dissolve residue in 8 M HNO₃ and allow to cool. This is the load solution for Sr determination (Appendix 1)



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APPENDIX 4

STRONTIUM DETERMINATION FOR LARGE VEGETATION, TISSUE AND MILK

Use double Sr resin column

Column Work

- _____ 10 mL 8 M HNO₃ (conditioning)
- _____ Load solution: 20 to 25 mL 8 M HNO₃
- _____ Rinse: 20 mL 8 M HNO₃
- _____ Rinse with additional 20 mL 8 M HNO₃ to remove excess potassium from column
- _____ Rinse: 20 mL 3 M HNO₃/0.05 M Oxalic Acid
- _____ Rinse: 10 mL 8 M HNO₃
- _____ **Elute:** 30 mL 0.05 M HNO₃ and catch in a c-tube (Record date and time)
- _____ Add 2 drops of Thymol Blue indicator and 6 M NaOH dropwise until blue
- _____ Add 3 mL 0.75 M sodium carbonate and heat in microwave until warm
- _____ Wait approximately 30 minutes
- _____ Filter



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VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF RADIUM-226

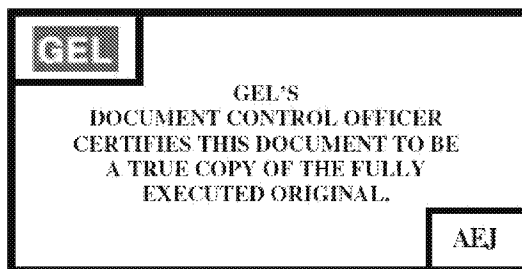
(GL-RAD-A-008 REVISION 15)

APPLICABLE TO METHOD:

EPA 600/4-80-032 Method 903.1 (Modified)
DOE EML HASL-300 Method Ra-04-RC (Modified)

PROPRIETARY INFORMATION

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF RADIUM-226**2.0 METHOD OBJECTIVE, PURPOSE, AND SUMMARY**

- 2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for Radium-226 in various matrices.
- 2.2 This method has been modified on the basis of GEL's Performance Based Measurement System (PBMS).
- 2.3 Solid matrices are decomposed by digestion in accordance with GL-RAD-A-015 for Digestion for Soil. The digestate is evaporated to dryness and diluted to known volume with nitric acid solution. A stream of nitrogen or helium gas is purged through the sample to initially remove radon from a water sample or solid sample digestate. The sample is then sealed and radon is allowed to ingrow. The radon, which is supported entirely by Ra-226 in the sample, is then purged with helium and trapped on a liquid nitrogen cold trap. The trap is sealed and warmed. The radon is then transferred by vacuum to a Lucas cell and counted after three hours in the cell.

3.0 METHOD SCOPE, APPLICABILITY AND DETECTION LIMIT

- 3.1 GEL Laboratories LLC (GEL) utilizes methods that are derived from established sources. This method has been modified from the source method EPA 600/4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," August 1980, Method 903.1, and uses the same principles of radiochemical concentration and counting. EPA Method 903.1 is written for drinking water. This method has been modified to accommodate various matrices as discussed in section 11.0.
- 3.2 Method Detection Limit (MDL): typical minimal detectable activity (MDA) for samples analyzed for Ra-226 is 1pCi/L or 1pCi/G.

4.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Team Leader. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

- 5.1 AlphaLIMS: The data system used at GEL Laboratories LLC.
- 5.2 Batch: Environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.
- 5.3 Deionized (DI) water: Type I DI water. Refer to GL-LB-E-016.
- 5.4 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.



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- 5.5 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
- 5.6 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.
- 5.7 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 5.8 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.
- 5.9 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

6.0 INTERFERENCES

The analysis of samples for Ra-226 content by Rn-222 emanation is very specific using this procedure, and the separation of radium from other elements is not required. Sample losses can occur only as the result of improper sample transfer. Due to the specific nature of Ra-226 measurement by this method, the use of stable barium carrier or radioactive Ba-133 tracer for yield monitoring is not required.

7.0 SAFETY PRECAUTIONS AND WARNINGS

- 7.1 Personnel performing this analytical procedure are trained to the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.
- 7.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.
- 7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for Handling Biological Materials.
- 7.4 If there is any question regarding the safety of any laboratory practice, stop immediately, and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

- 8.1 Apparatus and Equipment
- 8.1.1 De-emanation system with cold trap
- 8.1.2 Liquid nitrogen Dewars
- 8.1.3 1 Liter plastic bottles



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- 8.1.4 Amber latex tubing
- 8.1.5 Small tubing clamps
- 8.1.6 Lucas cells
- 8.1.7 Teflon beakers
- 8.1.8 HI-pore diffusers
- 8.1.9 Graduated cylinder 500 mL
- 8.1.10 Teflon watch glasses

8.2 Instrumentation

- 8.2.1 Radon flask counter with scalar

9.0 REAGENTS AND STANDARDS

9.1 Reagents

- 9.1.1 All chemicals should be of reagent grade or equivalent whenever they are commercially available.
- 9.1.2 Deionized water (DI): Type I water
- 9.1.3 Concentrated nitric acid (16 M HNO₃)
- 9.1.4 Boric acid, granular, A.C.S. grade
- 9.1.5 Liquid nitrogen (LN)

9.2 Standards

- 9.2.1 NIST traceable Ra-226 standard
- 9.2.2 Refer to GL-RAD-M-001 for instructions concerning the preparation of standard solutions.

10.0 SAMPLE HANDLING AND PRESERVATION

- 10.1 Soil samples require no preservation and may be shipped in any suitable container.
- 10.2 Water samples should be collected in plastic bottles and preserved with concentrated nitric acid to pH < 2.
- 10.3 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should adjust the pH with Nitric Acid to a pH < 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This acidification should be documented on a batch history sheet and attached to the batch paper work.

11.0 SAMPLE PREPARATION

NOTE: Aliquot size may be estimated by using the count time estimator spreadsheet.



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11.1 Solid matrices

NOTE: Alternatively, Ra-226 analysis in solid matrices can be completed as detailed in the Gamma Spectroscopy SOP, GL-RAD-A-013.

11.1.1 Transfer an appropriate aliquot (typically 1g) of sample to a labeled Teflon beaker. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced on the Queue sheet. Record all aliquots on the Queue sheet. The Blank and LCS aliquot should be recorded on the Queue sheet to be the same aliquot as the largest sample in the batch. Prepare the MB and LCS with a small amount of nitric acid.

NOTE: Unless the client provides a blank filter for this analysis, there is no appropriate material identified for the MB and LCS.

11.1.2 Add 0.1 mL of working Ra-226 standard to MS, MSD, LCS and LCSD as applicable. Record standard IDs and volumes on the Queue sheet.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing on the Queue sheet.

11.1.3 For analyses that require sample dissolution, digest solid samples as detailed in GL-RAD-A-015.

11.1.4 Dissolve the sample residue in 5 mL conc. nitric acid and transfer to a labeled de-emanation bottle. Dilute to 500 mL with DI water.

11.1.5 Proceed to Step 11.2.4 of this procedure.

11.2 Water samples

11.2.1 Transfer an appropriate aliquot (typically 500 mL) of sample to a labeled de-emanation bottle. Record aliquots on the Queue sheet.

NOTE: Each airtight cap should be placed on an empty nalgene bottle and checked for leaks before each use.

11.2.2 Prepare a MB and LCS using DI water and a small amount of Nitric acid to a pH<2. The volume should be the same as the largest volume of sample used in the batch and should be recorded on the Queue sheet.

11.2.3 Add 0.1 mL of working Ra-226 standard to MS, MSD, LCS and LCSD as applicable. Record standard IDs and volumes on the Queue sheet.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing on the Queue sheet.



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- 11.2.4 To remove radon from the sample, purge for at least 30 minutes with helium or nitrogen at a flow rate vigorous enough to remove radon from sample.
- 11.2.5 At the end of the degassing, seal the sample by connecting the inlet and outlet lines together. Record the date and time as the END INIT DEGAS DATE/TIME on the sample Queue sheet. Allow the sample to ingrow for a minimum of three days.
- NOTE:** Before proceeding to Step 11.2.6 it is advisable to begin acquiring background checks on the Lucas cells that will be used during the sample de-emanation process.
- 11.2.6 Fill the Dewars with liquid nitrogen. Lift the platform holding the Dewars to completely submerge the cold trap in LN. Allow the cold trap to equilibrate before proceeding.
- 11.2.7 Refer to Figure 1 for Operation of Radon Emanation Line. Connect the sample to lines V-3 and V-4 and ensure that the connections are secure. Turn valves V-3 and V-4 to the sample position. Bubbles should be visible as the helium is now purging the radon into the cold trap. Monitor the purging of the sample. Allow the helium to flow for approximately 15 minutes. Afterwards, record the date and time of the sample de-emanation as the END LN DE-EM DATE/TIME on the Queue sheet.
- 11.2.8 After approximately 15 minutes, turn valves V-5 and V-6 to the closed position. This will seal the cold trap that now contains the sample radon. The cold trap contains brass filings to create surface area for radon condensation. Turn valves V-3 and V-4 to the bypass position.
- 11.2.9 Connect the Lucas cell to the system. Pull a vacuum on the system by turning on the vacuum pump and opening valve V-7. With cold trap still under LN quickly open and close valve V-6 to remove excess helium.
- 11.2.10 Ensure that valves V-5 and V-6 are closed. Pull a vacuum on the system and the Lucas cell by turning on the vacuum pump and opening the valve V-7.
- 11.2.11 Close valve V-7 and turn off the vacuum pump. Check the system for leaks by observing the vacuum gauge for approximately 30 seconds. The vacuum gauge should hold vacuum at -20 to -30 psi. If the vacuum does not hold, notify your Group Leader or Team Leader.
- 11.2.12 Remove the LN Dewar and gently warm the trap with warm water and/or a hot air gun. The trap should feel warm to the touch before proceeding.



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11.2.13 Open valve V-6 to the vacuum system. Place hands on valves V-5 and V-6. Keep eyes on the vacuum gauge. Open valve V-5 and close valve V-6 just before the vacuum pressure goes to zero.

NOTE: The pressure will drop fast so be alert to the clockwise direction required to close valve V-6.

NOTE: If the zero (atmospheric pressure) is slightly passed the sample may still be counted. The analyst is trying to avoid creating a large positive pressure within the Lucas cell, which may cause the cell to leak or rupture.

11.2.14 Allow the radon to equilibrate in the line for approximately 30 seconds. Disconnect the Lucas cell and allow the radon daughters to equilibrate for a minimum of three hours before counting the cell. Place the cell in the counter for approximately 5 minutes before beginning the sample count.

11.2.15 Count each sample for a minimum of 15 minutes. Record the date and time the count is started, the count time and the gross counts observed as the START COUNT DATE/TIME on the Queue sheet.

NOTE: Sample activity levels can be verified using a re-transfer technique. This is done by taking a sample that has been previously transferred to a de-emanation bottle and repeating steps 11.2.4 through 11.2.15.

11.2.16 The Lucas cell should be cleaned as soon as possible after the sample count is completed. The Lucas cell cleaning apparatus is connected to the helium and the vacuum.

11.2.17 Connect the Lucas cell to the cleaning apparatus. Turn on the vacuum pump and the helium inlet valve. Turn on the relay to flush and evacuate the cell for at least two minutes.

11.2.18 Store the cell under a slightly positive helium pressure until the next use. The cell should be stored for a minimum of three hours prior to use for sample analysis.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

NOTE: Client contractual QC requirements override the requirements in this section.

12.1 Analyst and Method Verification Requirements

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Requirements

12.2.1 A Method Blank (MB) should accompany each batch of 20 or less samples. The reported value of the blank should be less than or equal to the CRDL (contract required detection limit).



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- 12.2.2 The tracer added to all samples issued to calculate the method recovery. The method recovery of all samples should be between 25-125% when compared to the reference standard.
- 12.2.3 A Matrix Spike (MS) should be run with each batch of 20 or less samples. The recovery of the MS should be between 75-125%.
- 12.2.4 A Duplicate sample should be run with each batch of 20 or less samples. The relative percent difference (RPD) between the actual sample and the QC DUP results should be less than or equal to 20% if both the sample and QC DUP results are greater than 5 times the LLD or 100% if either result is less than 5 times the LLD.
- 12.2.5 A Laboratory Control Sample (LCS) should be run with each batch of 20 or less samples. The recovery of the LCS should fall between 75-125%.
- 12.3 Actions required if the Quality Control Requirements Are Not Met
 - 12.3.1 If any of the QC criteria from 12.2.1 through 12.2.5 cannot be satisfied, the analyst should inform their group leader and initiate a Nonconformance Report as outlined in GL-QS-E-004.

13.0 INSTRUMENT, CALIBRATION, STANDARDIZATION AND PERFORMANCE

- 13.1 Ludlum Model 2000 operating voltage, plateau generation and standard deviation:
 - 13.1.1 Place a sealed Lucas Cell Ra-226 source of sufficient activity on the detector approximately 5 minutes before counting. Set the front panel discriminator to 50 volts. Count the source and record the counts.
 - 13.1.2 Step the front panel discriminator up in 50-volt increments and acquire counts at the increasing voltages up to 2000 volts and record counts. Plot the gross counts on the y-axis and the voltage on the x-axis and determine the "knee" of the plateau.
 - 13.1.3 The knee is determined by drawing straight lines along the rising slope and the plateau portions of the curve. The knee is the point where these two lines intersect. The operating voltage should be selected at 50 – 150 volts above the "knee."
 - 13.1.4 Put a copy of the plateau for model 2000 scaler/radon flask counter in the Ra-226 Calibration File.
 - 13.1.5 To determine the control limits (standard deviation), place a sealed Lucas Cell Ra 226 source of sufficient activity on the detector. Acquire twenty counts and record each count. If the operating voltage remains the same there is no need to establish new control limits.
 - 13.1.6 Put a copy of the counts and calculation of standard deviation in the Ra-226 Calibration File.
- 13.2 Calibration, cell constant, efficiency and verification of the Lucas Cell:



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- 13.2.1 The analyst should determine which cells need to be calibrated. Calibration is done annually or when a new Lucas cell is received. Give yourself at least 3 weeks before old calibration has expired. Calibration dates are stored in LIMS and are updated by a Group Leader or a Team Leader after the calibration is complete.
- 13.2.2 Each Lucas Cell needs to be given a two or three digit number (old numbers can be reused). With the first number to each cell will be the detector and rig it goes to. (For example, 120 will go into detector 1 and rig 1, 220 will go into detector 2 and rig 2.) Each lucas cell has one detector it can be counted on and one rig that it can be transferred on.
- 13.2.3 A background count is performed on each cell before every calibration and verification run and each count is recorded in the logbook.
- 13.2.4 Each counting cell is calibrated by spiking a 500 mL DI water sample with a known dpm of Ra-226 activity. The sample is carried through the entire procedure. The procedure is performed 3 separate times to each cell. Record each count.
- 13.2.5 Put information from the three runs in an excel spreadsheet to calculate cell constant, average and standard deviation. Standard deviation needs to be less than 10 % of the cell constant average. Put the Ra-226 cell constant spreadsheet in the Calibration File.
- 13.2.6 Each counting cell will be verified by spiking 500 mL of DI water with a known dpm of Ra-226 activity. Each verification sample is carried through the entire procedure. Acceptance criteria is $100\% \pm 25\%$.
- 13.2.7 After processing verification, put the spreadsheet in the Ra-226 Calibration File.
- 13.2.8 When calibration file is complete, the Group Leader or Team Leader will update the CELLEFF file to change the old cell efficiency to the new cell efficiency. The new calibration date will be updated in LIMS and should also be placed on the rig itself at this time.

14.0 ANALYSIS AND INSTRUMENT OPERATION

Refer to GL-RAD-I-007 for instrument operating instructions.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-007 for equipment and instrument maintenance.

16.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

Date recording, calculation and reduction takes place in accordance with GL-RAD-D-006.



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17.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

Data is reviewed and packaged in accordance with GL-RAD-D-003 for Data Review, Validation and Data Package Assembly.

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND DISPOSAL

Laboratory waste is disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

- 20.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, USEPA, Method 903.1, August, 1980.
- 20.2 Mathieu, G.G., Biscaye, P.E., Lupton, R.A. "A System for Measurement of Rn-222 at Low Levels in Natural Waters." Health Physics, Vol. 55, No.6, pp. 989-992. 1988.
- 20.3 Key, R.M., Brewer, R.L., Stockwell, J.H., Guinasso, N.L., Schink, D.R., "Some Improved Techniques for Measuring Radon and Radium in Marine Sediments and Seawater. Marine Chemistry, pp. 251-264. October 30, 1978.
- 20.4 Special thanks to Dr Bill Burnett and his associates at Florida State University for their help in building the radon de-emanation system.
- 20.5 EML procedures manual. HASL-300-Ed. 28, 1997, Ra-04-RC, Vol. 1.

21.0 HISTORY

Revision 13: Texas audit finding, updates made to comply with NELAC standard.

Revision 14: Updated water sample preservation to pH<2. Type II to type I DI water.

Revision 15: Added a NOTE: for verification of sample activity level using re-transfer technique.



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Determination of Radium-226

SOP Effective 6/5/92

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FIGURE 1

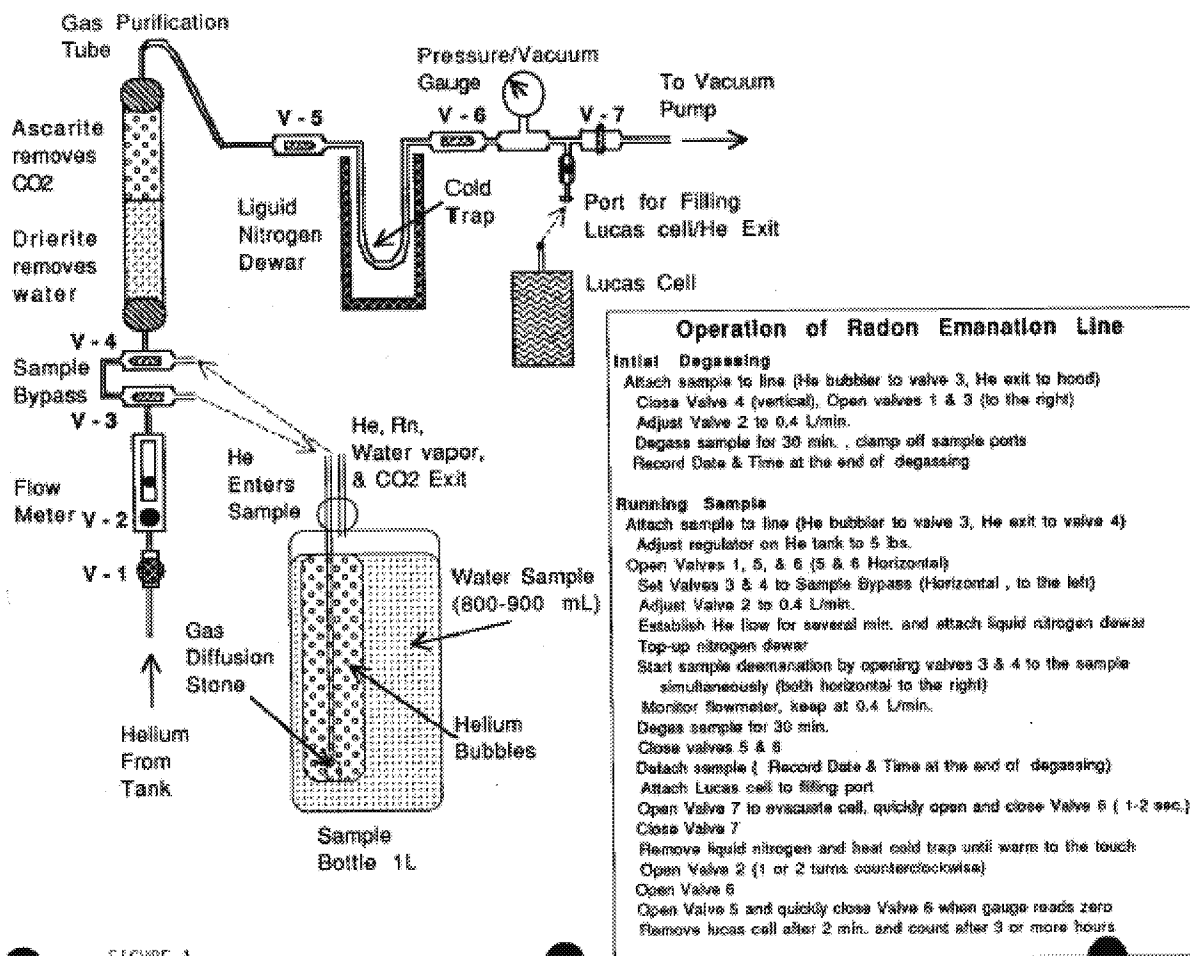


FIGURE 1

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VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

THE ISOTOPIC DETERMINATION

OF

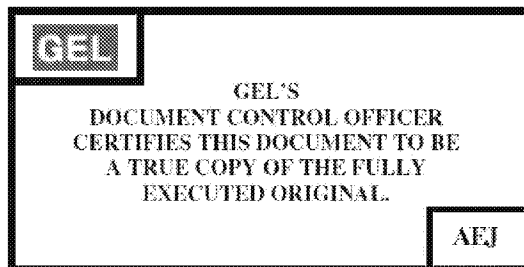
AMERICIUM, CURIUM, PLUTONIUM, AND URANIUM

(GL-RAD-A-011 REVISION 26)

APPLICABLE TO METHODS:
DOE RP800 1997 (Modified)
EML HASL-300 U-02-RC (Modified)
EML HASL-300 Am-05-RC (Modified)
DOE HASL-300 Pu-11-RC (Modified)
EPA SW-846 3050B (Modified)

PROPRIETARY INFORMATION

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1.0 STANDARD OPERATING PROCEDURE FOR THE ISOTOPIC DETERMINATION OF AMERICIUM, CURIUM, PLUTONIUM, AND URANIUM

2.0 METHOD OBJECTIVE, PURPOSE, CODE, AND SUMMARY

- 2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic americium, curium, plutonium, and uranium in a variety of liquid, filter and solid matrices. This method also gives specific guidance on determining U-232, Pu-242 and Am-243, which are typically used as isotopic tracers.
- 2.2 A soil sample is aliquoted and digested according to GL-RAD-A-015, if necessary. The elements are then separated through ion exchange resins. For liquid samples, transuranic elements are scavenged by coprecipitation with iron hydroxide. The precipitate is dissolved, and separation of elements is accomplished through ion exchange resins. The elements are then prepared for the measurement of radioactive isotopes by coprecipitation with neodymium fluoride. The neodymium fluoride precipitate is trapped on a filter, mounted on a metal disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.
- 2.3 This method has been modified from the source method from EML Methods Manual HASL-300 U-02-RC, Am-05-RC, and Pu-11-RC and uses similar principles of radiochemical separation and counting. Modifications include chemical separations utilizing Eichrom TEVA and TRU resins to facilitate separation of various elements. There are also variations in the concentrations of acids, as well as the application of these acids.
- 2.4 This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800, "Sequential Separation of Americium and Plutonium by Extraction Chromatography."
- 2.5 This method has been modified on the basis of GEL's Performance Based Measurement System (PBMS).
- 2.6 This method also contains a special procedure for digestion of samples in accordance with EPA method SW-846 3050B (Modified).

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

- 3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for Am/Cm/Pu/U is 1 pCi/L or 1 pCi/g for all isotopes.
- 3.2 Analyst training records are maintained as quality records as outlined in GL-QS-E-008. Analyst training and proficiency in the method is outlined in the Quality SOP for the Method Validation and Initial and Continuing Demonstrations of Capability, GL-QS-E-011.
- 3.3 Applicable matrices to this SOP are liquids, drinking water, vegetation, tissues, air filters, and solids.

NOTE: This method is not an EPA approved method for the analysis of drinking water.

4.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group or Team Leader. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

- 5.1 AlphaLIMS: Laboratory Information Management System used at GEL.
- 5.2 Batch: Environmental samples prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.
- 5.3 Deionized (DI) Water: Type I water, Refer to GL-LB-E-016.
- 5.4 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.
- 5.5 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
- 5.6 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.
- 5.7 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 5.8 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.
- 5.9 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.
- 5.10 Solid Reference Material (SRM): A solid material containing known and verified amounts of analytes.
- 5.11 Tracer: A known quantity of a radioisotope that is added to each sample of a chemically equivalent radioisotope of unknown concentration so that the yield of the chemical separation can be calculated.

6.0 INTERFERENCES

- 6.1 Internal tracer standards may have ingrown daughters that may interfere with the analysis. For example Th-228 will be present in aged U-232 standard, Fr-221 will be present in Th-229, which will interfere with the curium analysis, and U-232 will be present in Pu-236. These problems are overcome by running separate

aliquots of sample for thorium analysis, or by mathematical compensation for the interference.

- 6.2 Short lived radioactive progeny may ingrow on prepared filters. For example, the Ra-224 alpha peak will be present if the Th-228 parent is present. These interferences are minimized by counting sample as soon as possible after separation chemistry.

7.0 SAFETY PRECAUTIONS AND WARNINGS

- 7.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.
- 7.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.
- 7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.
- 7.4 If there is any regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

- 8.1 Apparatus and Equipment
- 8.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities
 - 8.1.2 Eichrom Technologies TEVA Resin, 100 – 150 µm particle size
 - 8.1.3 Eichrom Technologies TRU Resin, 100 – 150 µm particle size
 - 8.1.4 Vacuum pump and filtration apparatus
 - 8.1.5 Disposable filter funnels (containing 25 mm polypropylene filters with 0.1 µm pore size)
 - 8.1.6 Metal disks, 29 mm diameter
 - 8.1.7 Stainless steel tweezers
 - 8.1.8 Polypropylene centrifuge tube (50 mL)
 - 8.1.9 Sample drying and ashing apparatus
 - 8.1.10 Sample homogenization apparatus
 - 8.1.11 AG 1X8 anion exchange resin, 100 – 200 mesh
 - 8.1.12 Hot plate
 - 8.1.13 Beakers (Glass and Teflon of various sizes)
 - 8.1.14 2.5 cm³ column
 - 8.1.15 25 mL column funnel extension
 - 8.1.16 Watch glasses (various sizes)
 - 8.1.17 Digestion vessel
 - 8.1.18 Reflux cap

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- 8.1.19 Ribbed watch glass
- 8.1.20 Hot block
- 8.1.21 2.0 μm pore size plunger filter (PTF grade)

9.0 REAGENTS AND STANDARDS

9.1 Reagents

- 9.1.1 Neodymium carrier (500 mg/L)
- 9.1.2 Neodymium carrier (10,000 mg/L)
- 9.1.3 Carbon Colorant: Place four 47 mm cellulose nitrate filters in a beaker and add 5 mL concentrated sulfuric acid. Cover and heat on a hot plate with medium-high heat for approximately 2 to 4 hours. Cool dark residue completely. Slurry the residue in DI water and dilute to 1 L with DI water.
- 9.1.4 Hydrochloric acid (9 M HCl): Add 750 mL of concentrated hydrochloric acid to 100 mL of DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.5 Hydrochloric acid (3 M HCl): Add 250 mL of concentrated hydrochloric acid to 500 mL DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.6 Hydrochloric acid, concentrated (12 M HCl).
- 9.1.7 9 M Hydrochloric acid/0.05 M Ammonium iodide: Dissolve 7.24 g of ammonium iodide in 750 mL of concentrated hydrochloric acid and add to 100 mL of DI water. Allow to cool and dilute to 1 L with DI water. PREPARE DAILY.
- 9.1.8 Hydrochloric acid (6 M HCl): Add 500 mL of concentrated hydrochloric acid to 500 mL of DI water.
- 9.1.9 6 M Hydrochloric acid/0.52 M Hydrofluoric acid: Add 500 mL of concentrated hydrochloric acid and 18.6 mL of 49% hydrofluoric acid to 300 mL of DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.10 25% Hydrazine dihydrochloride: Dissolve 25 g of hydrazine dihydrochloride in 75 mL of DI water and dilute to 100 mL with DI water.
- 9.1.11 9 M Hydrochloric acid/0.04% Hydrogen peroxide: Add 8 drops of 30% hydrogen peroxide to 1 L of 9 M hydrochloric acid. PREPARE DAILY.
- 9.1.12 Ethyl alcohol (80% EtOH): Dilute 800 mL ethanol to 1 L with DI water.
- 9.1.13 Hydrochloric acid (0.1 M HCl): Add 8.3 mL of concentrated hydrochloric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.14 Hydrofluoric acid, concentrated (49% HF)
- 9.1.15 Hydrogen peroxide (30% H_2O_2)

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- 9.1.16 Iron Carrier (10 mg/mL): Dissolve 62.7 g of $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ or 72.3 g of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in 800 mL DI water and dilute to 1 L with DI water.
- 9.1.17 Nitric acid concentrated (16 M HNO_3)
- 9.1.18 Nitric acid (2 M HNO_3): Add 125 mL of concentrated nitric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.19 Nitric acid (1 M HNO_3): Dilute 62.5 mL concentrated nitric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.20 2 M Nitric acid/1 M Aluminum nitrate: Dissolve 375.13 g of aluminum nitrate nonahydrate, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, in 300 mL of DI water. Add 125 mL of concentrated nitric acid to the DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.21 Titanium (III) chloride, 10-20% reagent
- 9.1.22 Hydrochloric acid (2 M HCl): Add 167 mL of concentrated hydrochloric acid to 500 mL DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.23 Nitric acid (1 M HNO_3): Add 62.5 mL of concentrated nitric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.24 1.25 M Calcium nitrate: Dissolve 205 g of anhydrous calcium nitrate or 295 g hydrated calcium nitrate, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, in 500 mL of DI water. Dilute to 1L with DI water.
- 9.1.25 Phosphoric acid, concentrated (H_3PO_4)
- 9.1.26 Lanthanum (10,000 mg/L)
- 9.1.27 Sulfuric acid (0.1 M H_2SO_4): Add 5 mL concentrated sulfuric acid to 800 mL of DI water. Allow to cool and dilute to 900 mL.
- 9.1.28 Formic acid, concentrated
- 9.1.29 4 M Ammonium thiocyanate/0.1 M Formic acid: Add 60 g of ammonium thiocyanate and 1.0 mL of concentrated formic acid to a graduated cylinder and dilute to 200 mL with DI water. Prepare fresh daily.
- 9.1.30 1.5 M Ammonium thiocyanate/0.1 M Formic acid: Add 9.5 g of Ammonium thiocyanate and 0.5 mL of concentrated formic acid to a graduated cylinder and dilute to 100 mL with DI water. Prepare fresh daily.
- 9.1.31 Substrate suspension: Dilute 4 mL of neodymium chloride (10,000 mg/L), 80 mL of concentrated hydrochloric acid and 40 mL of carbon colorant to 1500 mL with DI water. Add 40 mL 49% hydrofluoric acid while swirling and dilute to 2 L with DI water.
- 9.1.32 Sulfuric acid, concentrated (18 M H_2SO_4).
- 9.1.33 Cellulose nitrate filters (47 mm)

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9.1.34 Ammonium hydroxide concentrated (14 N NH₄OH)

9.1.35 Acetone

9.2 Standards

9.2.1 NIST traceable standards: Am-241, Am-243, Cm-244, Pu-242, Pu-239, Pu-238, Pu-236, U-232, U-236, U-238.

9.2.2 Refer to GL-RAD-M-001.

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 Samples should be collected in a plastic bottle and preserved to approximately pH < 2 with nitric acid.

10.2 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should contact the Group Leader or Team Leader. If approved by the client, the analyst should adjust the pH with nitric acid to a pH < 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This acidification should be documented on a batch history sheet and attached to the batch paper work.

10.3 If the sample has exceeded the hold time the analyst should contact the Group Leader before continuing with the batch.

10.4 Soil and filter matrices require no preservation and may be shipped in any suitable container.

11.0 SAMPLE PREPARATION

NOTE: Aliquots may be estimated by using the count time estimator spreadsheet.

11.1 Soil Sample Preparation:

11.1.1 If not already done, dry and homogenize the sample by performing GL-RAD-A-021.

11.1.2 Measure an appropriate aliquot of soil (usually 0.2 g to 1.0 g) in a glass container or digestion vessel. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced on the Queue sheet. Record all aliquots on the Queue sheet. Add approximately 4 to 6 drops of iron carrier to the Blank and LCS beakers. The Blank and LCS aliquot should be recorded on the Queue sheet to be the same aliquot as the largest sample in the batch. For soils and other special matrices, such as vegetation, air filters, tissue, etc., Deionized water is a suitable matrix for use as the MB and LCS aliquot. Iron carrier may also be added to all samples in the batch, if necessary, based on the appearance and iron content of each sample. If Solid Reference Material is required, weigh out approximately 0.1 g into the LCS beaker and record the exact weight on the Queue sheet.

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- 11.1.3 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference batch Queue sheet and pull sheet for client requirements to determine appropriate tracer and spike.
- 11.1.3.1 For the determination of isotopic americium/curium, Am-243 is typically used as the tracer, and Am-241/Cm-244 are typically used as the spike.
- 11.1.3.2 For the determination of isotopic plutonium, Pu-242 is typically used as the tracer and Pu-239 is typically used as the spike. Pu-236 is an acceptable tracer provided no significant impurities are present.
- NOTE:** If Pu-241 is run in tandem, a separate MS and LCS is required to quantify Pu-241 spike recovery.
- 11.1.3.3 For the determination of isotopic uranium, U-232 is typically used as the tracer, and U-238 is typically used as the spike.
- NOTE:** The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing on the Queue sheet.
- 11.1.3.4 When running samples sequentially with Sr, all Sr carriers and spikes should be added prior to leach or digestion. See step 11.10.2 regarding collection procedures for Sr analysis.
- 11.1.4 If the analysis of the sample calls for quantification of U-232, Pu-242 or Am-243, the following steps shall be taken:
- 11.1.4.1 The sample will be run normally with the tracer indicated in sections 11.1.3 or 11.2.2.
- 11.1.4.2 A second run of the sample shall be made with a different tracer isotope such as U-236, Pu-236 or Cm-244. The quantification of the isotope that was normally the tracer can then be made. If there is any quantifiable activity a correction can be made to the initial run by calculating a correction ratio for the tracer recovery of the first run from the second run results.
- NOTE:** If prescribed to analyze by EPA method 3050B (Modified), proceed Appendix 7.
- 11.1.5 It is recommended that the samples be ashed in a muffle furnace as specified in GL-RAD-A-021B.
- 11.1.6 For uranium analysis, digest aliquot as specified in GL-RAD-A-015 and proceed to step 11.2.8.

- 11.1.7 A separate Am/Cm/Pu aliquot is treated with an aggressive acid leach of 6M or 9M hydrochloric acid depending on matrix and sample aliquot as described in the following steps. Uranium should not be run by this leaching technique.

NOTE: Determining the leaching routine is based on analyst experience with the matrix. The concentration required to obtain the leached sample will vary depending on the type of material, size of aliquot, muffling of sample, and other factors. The influence of these factors generally can be established by good judgment and experience with the materials being tested.

- 11.1.7.1 Place the sample in a beaker and add approximately 10 to 20mL of appropriate hydrochloric acid concentration per gram of sample with a minimum of 10 mL.
 - 11.1.7.2 Heat the samples on medium heat and cover with a watch glass. Allow to leach for a minimum of 2 hours. Agitate the sample periodically to enhance the leaching process.
 - 11.1.7.3 Allow the sample to partially cool and transfer to a centrifuge tube. Centrifuge the sample to separate the solid and leached portions.
 - 11.1.7.4 Decant the leachate to a clean labeled beaker, and rinse the solid phase with DI water. Centrifuge the sample and decant the leachate into the beaker.
 - 11.1.7.5 Evaporate the solution to dryness on medium heat.
 - 11.1.7.6 Proceed to step 11.2.8.
- 11.2 Aqueous Sample Preparation:
- 11.2.1 Add an appropriate aliquot of sample to a labeled beaker. Prepare a Blank and LCS using DI water and a small amount of concentrated nitric acid to a pH < 2. The volume of DI water used should be the same as the largest volume of sample in the batch. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced on the Queue sheet. Record all aliquots on the Queue sheets.
 - 11.2.2 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference batch Queue sheet and pull sheet for client requirements to determine appropriate tracer and spike.
 - 11.2.2.1 For the determination of isotopic americium/curium, Am-243 is typically used as the tracer, and Am-241/Cm-244 are typically used as the spike.
 - 11.2.2.2 For the determination of isotopic plutonium, Pu-242 is typically used as the tracer, and Pu-239 is typically used as the spike. Pu-

236 is an acceptable tracer, provided no significant impurities are present.

NOTE: If Pu-241 is run in tandem, a separate MS and LCS is required to quantify Pu-241 spike recovery.

11.2.2.3 For the determination of isotopic uranium, U-232 is typically used as the tracer, and U-238 is typically used as the spike.

11.2.2.4 When running samples sequentially with Sr, all Sr carriers and spikes should be added prior to initial iron precipitation to scavenge actinides. See section 11.10 regarding collection procedures for Sr analysis.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing on the Queue sheet.

11.2.3 If the analysis of the sample calls for quantification of U-232, Pu-242 or Am-243, the following steps shall be taken:

11.2.3.1 The sample will be run normally with the tracer indicated in sections 11.1.3 or 11.2.2.

11.2.3.2 A second run of the sample shall be made with a different tracer isotope such as U-236, Pu-236 or Cm-244. The quantification of the isotope that was normally the tracer can then be made. If there is any quantifiable activity a correction can be made to the initial run by calculating a correction ratio for the tracer recovery of the first run from the second run results.

11.2.4 If samples contain large amounts of sediment that the client requires analyzed with the liquid portion of the sample, proceed to step 11.9.

NOTE: Other sample matrices, such as vegetation, air filters, tissue, etc. are prepared as outlined in GL-RAD-A-026. The analyst must ensure that the appropriate tracer(s) are added to these other matrices as discussed in sections 11.1.3 or 11.2.2.

11.2.5 Add 1 mL of iron carrier (10 mg/mL).

11.2.6 Add concentrated ammonium hydroxide until turbidity persists, or pH > 9. Then add approximately 2 mL in excess. Heat to boiling for approximately 10 minutes or until precipitate breaks into fine particles. Allow to settle and cool.

11.2.7 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate.

NOTE: Exercise care in this step because finely divided material that contains the actinides may also be present in addition to the large iron hydroxide flocks.

- 11.2.8 Dissolve the precipitate from step 11.2.7 or residue from step 11.1.6 or 11.1.7.5 in 10 to 15 mL of 9 M hydrochloric acid /0.04% hydrogen peroxide solution.

NOTE: Samples may be dissolved with 10 to 15 mL of 9 M hydrochloric acid and then add 1 drop of 30% hydrogen peroxide as an alternative to dissolving with 9 M hydrochloric acid /0.04% hydrogen peroxide. This may also be done by dissolving samples with 10 to 15 mL 9 M hydrochloric acid and adding approximately 1 mL of DI water to approximately 1 mL of 30% hydrogen peroxide, mixing and adding one drop to each sample.

NOTE: If uranium only is required, the load solution is 10 to 15 mL of 9 M hydrochloric acid.

- 11.2.9 Slurry AG 1x8 anion resin (Cl form 100-200 mesh) in a squirt bottle with DI water. Transfer the resin to a small column to obtain a settled resin bed of approximately 2.5 mL.
- 11.2.10 Condition the column with 10 mL of 9 M hydrochloric acid.
- 11.2.11 Pass the sample solution from step 11.2.8 through the column and collect the eluate in a labeled, disposable 50 mL centrifuge tube for americium/curium analysis.
- 11.2.12 Rinse the column with 5 mL of 9 M hydrochloric acid and collect with the americium/curium fraction. Proceed to step 11.3.
- 11.2.13 Rinse the column with an additional 15 mL of 9 M hydrochloric acid and collect in a drip pan for disposal.
- 11.2.14 Elute plutonium by adding 10 mL of 9 M hydrochloric acid /0.05 M ammonium iodide solution, catching the plutonium elution in a labeled, disposable 50 mL centrifuge tube. Proceed to step 11.5 for plutonium microprecipitation for alpha spectroscopy. This elution may be omitted if plutonium analysis is not required.
- 11.2.15 Rinse the column with 15 mL of 6 M hydrochloric acid /0.52 M hydrofluoric acid and collect in a drip pan for disposal.
- 11.2.16 Rinse the column with 5 mL of 6 M hydrochloric acid and collect in a drip pan for disposal.
- 11.2.17 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute uranium from the column using 15 mL of 0.1 M hydrochloric acid. Proceed to step 11.6 for uranium microprecipitation for alpha spectroscopy.

11.3 Americium/Curium Separation via TRU Resin:

NOTE: If sample aliquot is small or liquid sample is clean and free of particulates continue with step 11.3.1 and TRU column work. If not, proceed to step 11.3.5 for additional clean-up steps and TRU column work.

- 11.3.1 Precondition a 2 mL TRU column with 5 mL of 9 M hydrochloric acid.

- 11.3.2 Pass the sample solution from step 11.2.12 through the column collecting in a drip pan for disposal.
- 11.3.3 Rinse the column with 5 mL of 9 M hydrochloric acid collecting in a drip pan for disposal.
- 11.3.4 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute americium and curium from the column using 20 mL of 3 M hydrochloric acid. Proceed to step 11.4 Americium/Curium microprecipitation for alpha spectroscopy.
- 11.3.5 Add 0.5 mL of 1.25 M calcium nitrate to the elution from step 11.2.12.
- 11.3.6 Add 1.0 mL of phosphoric acid. Swirl to mix.
- 11.3.7 Dilute to approximately 30 mL with DI water.
- 11.3.8 Add 28-30% ammonium hydroxide to pH of 8 to 10 to precipitate calcium phosphate. Do not over precipitate.
- 11.3.9 Allow to cool then spin samples in a centrifuge and pour off supernate.
- 11.3.10 Add approximately 25 mL of DI water to centrifuge tube, cap, and shake vigorously to break up precipitate.
- 11.3.11 Spin samples in a centrifuge and pour off supernate.
- 11.3.12 Add 15 mL of 2 M nitric acid/1 M aluminum nitrate to centrifuge tube and dissolve precipitate. Gently heat if necessary. Solution should be clear.
- 11.3.13 Precondition a 2 mL TRU column with 10 mL of 2 M nitric acid, collecting the rinse in a drip pan for disposal.
- 11.3.14 Pass the sample solution from step 11.3.12 through the column, collecting the load solution in a drip pan for disposal.
- 11.3.15 Rinse the column twice with 5 mL of 2 M nitric acid and collect the rinse in a drip pan for disposal.
- 11.3.16 Rinse the column with 5 mL of 1 M nitric acid and collect the rinse in a drip pan for disposal.
- 11.3.17 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute americium and curium from the column using 20 mL of 3 M hydrochloric acid. If rare earth elements are suspected in the sample proceed to step 11.8.1 to separate rare earth elements via TEVA resin, otherwise, continue with step 11.4.
- 11.4 Americium/Curium Microprecipitation:
 - 11.4.1 Dilute americium elution from step 11.3.4, 11.3.17, or 11.8.11 to approximately 40 mL with DI water. Add 0.1 mL of neodymium carrier (500 mg/L) to the solution and swirl to mix. Add 5 mL of 49% hydrofluoric acid and swirl to precipitate fluorides. Allow solution to sit for approximately 30 minutes, then proceed to step 11.7.1.

11.5 Plutonium Microprecipitation

11.5.1 Dilute plutonium elution from step 11.2.14 to approximately 40 mL with DI water. Add 0.1 mL of neodymium carrier (500 mg/L) and swirl. Add approximately 3 to 4 drops of 25% hydrazine dihydrochloride and swirl to mix. Let the solution sit for approximately 10 minutes, and add 5 mL of 49% hydrofluoric acid. Swirl to mix. Allow solution to sit for approximately 30 minutes, then proceed to step 11.7.1.

11.6 Uranium Microprecipitation:

11.6.1 Dilute uranium elution from step 11.2.17 to approximately 40 mL with DI water. Add 0.1 mL of neodymium carrier solution (500 mg/L) and swirl to mix. Add 0.5 mL of titanium (III) chloride solution and allow the sample to sit for approximately 30 seconds. Add 5 mL of 49% hydrofluoric acid to precipitate fluorides. Allow the solution to sit for approximately 30 minutes, then proceed to step 11.7.1.

11.7 Sample Filtration:

11.7.1 Place a disposable filter funnel on the filter support screen. Wet the filter with 80% ethyl alcohol and apply vacuum.

11.7.2 Add 5 mL of substrate suspension. After solution has passed through filter, add another 5 mL of substrate suspension.

11.7.3 Add 1 mL of the carbon colorant.

11.7.4 Filter the fluoride precipitated solution through the filter paper. Rinse the centrifuge tube with approximately 5 mL DI water and pass through filter.

11.7.5 Rinse the funnel with 80% ethyl alcohol.

CAUTION: Directing a stream of liquid onto the filter will disturb the distribution of the precipitate on the filter and render the sample unsuitable for alpha spectrometry resolution.

11.7.6 Without turning off the vacuum, remove the funnel.

11.7.7 Turn off vacuum and remove filter. Mount filter on a labeled 29 mm flat planchet. Ensure that the filter is centered and as flat as possible on the planchet.

NOTE: Care should be taken not to touch the active area of the filter with tweezers.

11.7.8 Place the mounted filter under a heat lamp for approximately 5 minutes or allow to air dry completely prior to alpha spectrometry measurement.

11.7.9 Submit samples for Alpha Spec counting.

NOTE: After Alpha Spec counting and review is complete, if Pu-241 analysis is required, proceed to SOP GL-RAD-A-035 step 11.2.31.

11.8 Separation of Americium from the Rare Earth Elements via TEVA Resin:

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|--|--|

- 11.8.1 Transfer the elution from Step 11.3.17 to a clean beaker and add 0.3 mL of lanthanum. Gently cook dry on low heat.
 - 11.8.2 Once the samples have cooled, add 5 mL of concentrated nitric acid and approximately 2 mL of 30% hydrogen peroxide. Heat on a hot plate at low heat to dryness. Cool and repeat.
 - 11.8.3 Dissolve residue in approximately 1 mL of 0.1 M sulfuric acid. Evaporate until a very small amount of acid remains.
 - 11.8.4 Dissolve residue in approximately 1 mL of concentrated formic acid. Evaporate until a very small amount of acid remains.
 - 11.8.5 Repeat step 11.8.4, and evaporate the sample under low heat until the beaker is gently dry.
 - 11.8.6 Dissolve the sample in 10 mL of 4 M ammonium thiocyanate/0.1 M formic acid. Be sure that the 4 M ammonium thiocyanate/0.1 M formic acid is prepared fresh daily.
 - 11.8.7 Condition a TEVA column with 5 mL of 4 M ammonium thiocyanate/0.1 M formic acid, collecting the rinse in a drip pan for disposal.
 - 11.8.8 Load the sample onto the TEVA column, collecting the load in a drip pan for disposal.
 - 11.8.9 Rinse the beaker with 5 mL of 4 M ammonium thiocyanate/0.1 M formic acid and add to the column, collecting the rinse in a drip pan for disposal.
 - 11.8.10 Rinse lanthanum and other rare earth elements from the column with 10 mL of 1.5 M ammonium thiocyanate/0.1 formic acid, collecting the rinse in a drip pan for disposal. Be sure that the 1.5 M ammonium thiocyanate/0.1 formic acid is prepared fresh daily.
 - 11.8.11 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute americium with 20 mL of 2 M hydrochloric acid.
 - 11.8.12 Proceed to Step 11.4 to precipitate and filter samples.
- 11.9 Samples Containing Large Amounts of Sediment
- NOTE:** When aliquoting samples that contain large amounts of sediment, ensure that the sample is thoroughly homogenized.
- 11.9.1 Evaporate to dryness on medium to low heat.
 - 11.9.2 Muffle in a furnace at approximately 550° C for a minimum of 2 hours.
 - 11.9.3 If uranium analysis is required, leach for approximately 30 minutes and proceed to step 11.1.6.
 - 11.9.4 If americium, curium, or plutonium analyses are required, proceed to step 11.1.7.

NOTE: Samples requiring americium extraction **MUST** be separated from rare earth elements via TEVA resin.

11.10 Preparation technique for Sr samples run in tandem with americium, plutonium, or uranium analysis.

11.10.1 Supernate from step 11.2.7 should be decanted into a clean, labeled beaker for further Sr analysis. Do not discard.

11.10.2 Elution from steps 11.2.11 and 11.2.12 should be collected in a clean, labeled centrifuge tube.

11.10.3 If americium/curium analysis is not required sample should be combined with supernate from step 11.10.1. Then proceed to appropriate SOP for Sr analysis.

11.10.4 If americium/curium analysis is needed proceed to step 11.3. Elution from steps 11.3.2 and 11.3.3 should be collected in a clean, labeled centrifuge tube. Elution should be combined with supernate from step 11.10.1. Then proceed to appropriate SOP for Sr analysis.

12.0 QUALITY CONTROL REQUIREMENTS

NOTE: Client contractual QC requirements override the requirements in this section.

12.1 Analyst and Method Verification Requirements

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Requirements

12.2.1 A method blank (MB) should accompany each batch of 20 or less samples. The reported value of the blank should be less than or equal to the contract required detection limit (CRDL).

12.2.2 The tracer added to all samples is used to calculate the method recovery. The method recovery of all samples should be between 15-125% when compared to the reference standard.

12.2.3 A duplicate (DUP) sample should be run with each batch of 20 or less samples. The relative percent difference (RPD) between the actual sample and the DUP should be less than or equal to 20% if both the sample and DUP results are greater than 5 times the minimal detectable concentration (MDC), or 100% if they are both less than 5 times MDC. If both results are less than the MDC, then limits are not applicable.

12.2.4 A laboratory control sample (LCS) should be run with each batch of 20 or less samples. The recovery of the LCS should fall between 75-125%.

12.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the QC criteria from 12.2.1 through 12.2.4 cannot be satisfied, the analyst should inform the Group Leader and initiate a Data Exception Report (DER) as outlined in GL-QS-E-004.

13.0 INSTRUMENT CALIBRATION AND PERFORMANCE

For direction on calibration and instrument performance refer to GL-RAD-I-009.

14.0 ANALYSIS AND INSTRUMENT OPERATION

For analysis and instrument operation refer to GL-RAD-I-009.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

For maintenance of system refer to GL-RAD-I-010.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation, and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Refer to GL-RAD-D-003 for instructions concerning the data review process, approval, and transmittal.

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

- 20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.
- 20.2 EML Procedures Manual HASL-300, Volume I February 2000, Method U-02-RC, Revision 1.
- 20.3 DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800, "Sequential Separation of Americium and Plutonium by Extraction Chromatography."
- 20.4 Analytical Chemistry. Rapid Determination of Thorium-230 in Mill Tailings by α Spectrometry. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol. 59, No. 21, 1987.
- 20.5 Los Alamos Health and Environmental Chemistry: Analytical Techniques. LA-10300-M Vol. 1, September 1987.
- 20.6 Special thanks to Dr. Bill Burnett and his associates for assistance in developing this method at Florida State University.
- 20.7 EML Procedures Manual HASL-300, Volume II February 1997, Method Am-05-RC.
- 20.8 U.S. Department of Energy, Environmental Measurements Laboratory Procedures Manual HASL-300, Section 4.5.4, Vol. 1, Pu-11-RC, 28th Ed., 1997

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21.0 HISTORY

Revision 26: Added Appendix 7 to include steps of digestion of soil using EPA Method 3050B (Modified).

Revision 25: 1 mL of Titanium chloride to 0.5 mL in sample preparation section and checklist.

Revision 24: Updated Appendix 1. Combined steps 11.3.18 with step 11.3.17 for clarification.

Revision 23: Added DOE HASL-300 Pu-11-RC (modified) to Applicable methods on title page. Section 2.3 added Pu-11-RC as a source method. Updated reagent section. Added U-236 to section 9.2.1. Added section 11.1.4 regarding tracers. Note added after section 11.2.2.2 regarding Pu-241 analysis if run in tandem. Omitted section 11.2.3.2 and 11.2.3.3. Removed plutonium cookdown from sections 11.2.14 and 11.5. Removed MS requirement from section 12.2. Updated section 16.0.

Revision 22: Added step 11.2.2.4 and section 11.10.

Revision 21: Changed stainless steel to metal in sections 2.2 and 8.1.6.

Revision 20: Updated reagent section. Notes added after section 11.1.3.2 and 11.7.9 regarding Pu-241 analysis if run in tandem. Omitted cookdown procedure for Americium and Uranium. Updated sections 11.4 and 11.6 microprecipitation steps. Added Appendix 6: Sample Cleanup from an Alpha Spec Filter.

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APPENDIX 1

AMERICIUM, CURIUM, PLUTONIUM, AND URANIUM

Use a 2.5 cm³ column with 1X8 anion resin (Cl⁻ form 100-200 mesh)

COLUMN WORK

- _____ 10 mL 9 M HCl (Conditioning)
- _____ Load solution: 10 to 15 mL 9 M HCl / 0.04% H₂O₂ (Catch in C-Tube for Am/Cm)
NOTE: If U only is required the load solution is 10 to 15 mL of 9 M HCl
- _____ 5 mL 9 M HCl (Catch in C-Tube for Am/Cm then proceed to Appendix 2 or 3 as appropriate for Am/Cm procedure)
- _____ 15 mL 9 M HCl (Rinse)
- _____ **Elute** Pu: 10 mL 9 M HCl / 0.05 M NH₄I (Catch in C-Tube then proceed to Appendix 4 Plutonium Precipitation)
- _____ 15 mL 6 M HCl / 0.52 M HF (Rinse)
- _____ 5 mL 6 M HCl (Rinse)
- _____ **Elute** U: 15 mL 0.1 M HCl (Catch in C-Tube)
- _____ Proceed to Appendix 4 for Uranium Precipitation

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APPENDIX 2

AMERICIUM / CURIUM CONTINUATION

AMERICIUM / CURIUM

- _____ 0.5 mL 1.25 M Calcium nitrate
- _____ 1.0 mL Phosphoric acid and swirl
- _____ Dilute to approximately 30 mL with DI water
- _____ Concentrated NH_4OH to pH of 8 to 10
- _____ Centrifuge samples and pour off supernate
- _____ Add approximately 25 mL DI water and shake samples to break up precipitate
- _____ Centrifuge samples and pour off supernate
- _____ 10 mL 2 M HNO_3 (Condition 2 mL TRU Resin Column)
- _____ Load Solution: 15 mL 2 M HNO_3 / 1 M $\text{Al}(\text{NO}_3)_3$
- _____ 5 mL 2 M HNO_3 (Rinse)
- _____ 5 mL 2 M HNO_3 (Rinse)
- _____ 5 mL 1 M HNO_3 (Rinse)
- _____ **Elute** Am/Cm: 20 mL 3 M HCl (Catch in C-Tube)
- _____ Proceed to Appendix 4 for Am/Cm precipitation

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APPENDIX 3

AMERICIUM / CURIUM CONTINUATION

AMERICIUM / CURIUM

- ___ 5 mL 9 M HCl (Condition 2 mL TRU Resin Column)
- ___ Load solution from Appendix 1 (catch in drip pan)
- ___ 5 mL 9 M HCl (Rinse)
- ___ **Elute** Am/Cm: 20 mL 3 M HCl (Catch in C-tube)
- ___ Proceed to Appendix 4 for Am/Cm precipitation

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APPENDIX 4

AMERICIUM / CURIUM PRECIPITATION

_____ Dilute elution with DI water to approximately 40 mL

_____ 0.1 mL 500 mg/L Neodymium and swirl

_____ 5 mL 49% HF and swirl

_____ Wait approximately 30 minutes

_____ Filter

PLUTONIUM PRECIPITATION

_____ Dilute elution with DI water to approximately 40 mL

_____ 0.1 mL 500 mg/L Neodymium and swirl

_____ Approximately 3 to 4 drops 25% Hydrazine dihydrochloride and swirl

_____ Wait approximately 10 minutes

_____ 5 mL 49% HF and swirl

_____ Wait approximately 30 minutes

_____ Filter

URANIUM PRECIPITATION

_____ Dilute elution with DI water to approximately 40 mL

_____ 0.1 mL 500 mg/L Neodymium and swirl

_____ 0.5 mL Titanium chloride and swirl

_____ Wait approximately 30 seconds

_____ 5 mL 49% HF and swirl

_____ Wait approximately 30 minutes

_____ Filter

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APPENDIX 5

RARE EARTH CLEAN-UP

- _____ Transfer elution from TRU column to a clean beaker and add 0.3 ml of lanthanum
- _____ Evaporate to dryness on low heat
- _____ 5 mL concentrated HNO₃ and approximately 2 mL of 30% H₂O₂. Evaporate to dryness on low heat.
- _____ 5 mL concentrated HNO₃ and approximately 2 ml of 30% H₂O₂. Evaporate to dryness on low heat.
- _____ Approximately 1 mL of 0.1 M Sulfuric Acid. Evaporate to dryness on low heat
- _____ Approximately 1 mL of concentrated Formic Acid. Evaporate to dryness on low heat
- _____ Approximately 1 mL of concentrated Formic Acid. Evaporate to dryness on low heat
- _____ 5 mL 4 M Ammonium thiocyanate/0.1 M Formic acid (Condition 2 mL TEVA column)
- _____ Load Solution: 10 mL 4 M Ammonium thiocyanate/0.1 M Formic acid
- _____ 5 mL 4 M Ammonium thiocyanate/0.1 M Formic acid (Rinse)
- _____ 10 mL 1.5 M Ammonium thiocyanate/0.1 M Formic acid (Rinse)
- _____ **ELUTE** Am: 20 mL of 2 M HCl (catch in C-tube)
- _____ Proceed to Appendix 4 for Am/Cm precipitation

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APPENDIX 6

SAMPLE CLEANUP FROM AN ALPHA SPEC FILTER

1. Remove the filter from the mounting disc by wetting the filter with a small amount of acetone and pulling the filter off the disc using tweezers. Place the filter in a labeled small glass beaker.
2. Add 4-6 drops of iron carrier (10 mg/L), 10 mL of concentrated hydrochloric acid and 1.0 mL of 5% boric acid solution.
3. Fill the bulb end of a disposable pipette with DI water and turn upside down. Place on the filter in the glass beaker to ensure the filter remains submerged.
4. Heat on a hot plate for 30 minutes frequently stirring the filter, flipping it over then back etc. Use the disposable pipette to stir and flip.
5. Remove the filter from the solution and rinse with DI water. Do this over the glass beaker so the DI water rinse falls back into the beaker.
6. Evaporate to dryness.
7. Proceed to section 11.2.8 and perform separations as specified.

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APPENDIX 7

SPECIAL PROCEDURE: DIGESTION OF SOILS AND SEDIMENTS BY EPA METHOD 3050B **(MODIFIED)**

- Complete Steps 11.1.1 thru 11.1.4 of this procedure
- Add 5 mL of [HNO₃] and 5 mL of Type I DI water to the samples and quality control samples
- Gently swirl the sample and acid mixture
- Cover the sample with a reflux cap or watch glass and heat the sample in a hot block at 95° +/- 5° C. Reflux the sample for 10 to 15 minutes
- Remove the samples from the hot block and allow the samples to cool
- Add 5 mL of [HNO₃], return samples to hot block, replace the watch glass, reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by nitric acid, add an additional 5 mL [HNO₃] until no brown fumes are given off by the sample.
- Using a ribbed watch glass or reflux cap, allow the solution to evaporate to approximately 5 mL, without boiling, or heat for 2 hours.
- Remove the sample from the hot block and allow to cool
- Add 2 mL of Type I DI water and 3 mL of 30% H₂O₂, return samples to hot block and allow the peroxide reaction to occur. Continue to add H₂O₂ in 1 mL increments until effervescence subsides. Do not add more than 10 mL of H₂O₂.
- Cover the samples with ribbed watch glass or reflux cap and heat the samples at 95° +/- 5° C for 2 hours, without boiling.
- Remove from hot block and allow the samples to cool
- Cap the samples and shake well
- Filter each sample with a 2.0 µm pore size plunger type filter (PTF grade) or allow to settle overnight.
- Transfer liquid phase to clean labeled centrifuge tube
- Proceed to Step 11.2.6 of this procedure

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VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

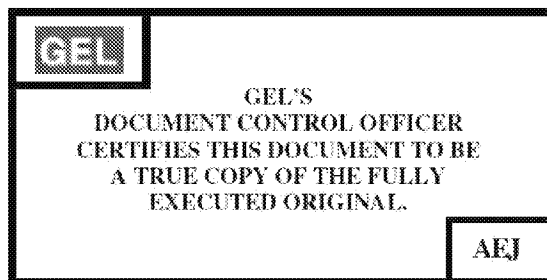
**STANDARD OPERATING PROCEDURE
FOR
THE DETERMINATION
OF
GAMMA ISOTOPES**

(GL-RAD-A-013 REVISION 26)

APPLICABLE TO METHODS:
EPA 600/4-80-032 Method 901.1
DOE EML HASL-300 Section 4.5.2.3
DOE EML HASL-300 Ga-01-R

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF GAMMA ISOTOPES

2.0 METHOD OBJECTIVE, PURPOSE, AND SUMMARY

- 2.1 This standard operating procedure (SOP) provides the necessary instructions to conduct the analysis for gamma isotopes in water, soil, urine, filters, drinking water and miscellaneous matrices.
- 2.2 Water samples are typically counted in Marinelli beakers. Soil samples are typically sealed in aluminum cans, which can be counted immediately if Ra-226 is not desired. If Ra-226 is desired, the sealed can is set aside for minimum of 20 days to allow equilibrium between Rn-222 and Bi-214 to become re-established. Ra-226 is then quantified using the 609 keV line of Bi-214.
- 2.3 This method is based on the source method EPA 600/4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," August 1980, Method 901.1, and the Department of Energy (DOE) EML Procedures Manual source method for Gamma PHA in environmental samples, HASL-300 Section 4.5.2.3 and Ga-01-R, Gamma Radioassay.
- 2.4 This SOP is applicable for analyzing samples that contain radionuclides emitting gamma photons with energies ranging from about 5 to 2000 keV (including I-131).

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

- 3.1 Minimum Detectable Activity (MDA): The MDA is based upon sample volume, Compton background, instrument efficiency, count time, and other statistical factors, as well as specific isotopic values such as abundance and half-life. A typical detection limit is 10 pCi/L or 0.1 pCi/g (based on Cs-137). The MDA for drinking water samples is 10 pCi/L (based on Cs-137).
- 3.2 Method Precision: Typical Relative Percent Difference (RPD) is 20% or less or 100% or less if the activity is less than five times the MDA.
- 3.3 Method Bias (Accuracy): The method accuracy requirement for gamma, measured by running a Laboratory Control Sample (LCS) with each batch, is 25% of the true value. For drinking water samples, laboratory fortified blanks (LFB, equivalent to LCS) recoveries should be between 90-110% of the known value.
- 3.4 Procedures contained in this SOP may be used to analyze REMP samples.
- 3.5 Analysts training records are maintained as quality records as outlined in GL-QS-E-008. Analysts training and proficiency in the method is outlined in the Employee Training SOP GL-HR-E-002.
- 3.6 For drinking water samples, analyst initial and ongoing demonstrations of proficiency will follow critical elements for radiochemistry, chapter VI, section



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1.5, of The Manual for the Certification of Laboratories Analyzing Drinking Water (reference 20.5).

- 3.7 Sensitivity studies will follow critical elements for radiochemistry, chapter VI, section 7.3 of The Manual for the Certification of Laboratories Analyzing Drinking Water (reference 20.5).

4.0 METHOD VARIATIONS

- 4.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Team Leader. Variations to a method will be documented with the analytical raw data.
- 4.2 Filter samples can either be counted directly, or digested prior to counting. If filters are digested, they are digested in accordance with GL-RAD-A-026.
- 4.3 No method modifications are permitted for drinking water samples.

5.0 DEFINITIONS

- 5.1 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.
- 5.2 Deionized (DI) water: Type I water. Refer to GL-LB-E-016.
- 5.3 AlphaLIMS: GEL's Laboratory Information Management System.
- 5.4 Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.
- 5.5 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.
- 5.6 Laboratory Duplicate (DUP): For soils, when sufficient sample is available, a separate duplicate will be prepared. For liquid samples and when sufficient sample is not available for solids, an independent count of the sample container will be performed to show precision.
- 5.7 Laboratory Control Sample (LCS): A sample matrix, similar to the batch of associated samples (when available) that is free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. The LCS is equivalent to a Fortified Blank in the EPA drinking water compliance manual (See to section 20.5).
- 5.8 Refer to SOP GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.



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6.0 INTERFERENCES

- 6.1 Some gamma isotopes emit gamma lines that may overlap with other isotopes. If the energies of the two isotopes are within the energy tolerance setting, the peaks may not be resolvable and may give a positive bias to the result. This problem is minimized by careful review of the peak search.
- 6.2 Soil samples may vary in density from the standard used for calibration. A density correction is applied to the "CAN" geometry. This correction was determined using solids with weights varying between 54 g and 192 g.

7.0 SAFETY PRECAUTIONS AND WARNINGS

- 7.1 Keep hands free from moving parts of canning device and gamma shields.
- 7.2 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.
- 7.3 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.
- 7.4 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.
- 7.5 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

- 8.1 Ancillary Equipment
 - 8.1.1 100 cc aluminum cans with lids for soil and miscellaneous samples
 - 8.1.2 10 cc Gelman Sciences Petri dish for soil, filters and miscellaneous samples
 - 8.1.3 2 L and 500 mL Marinelli beakers for water samples
 - 8.1.4 Air displacement pipettes
 - 8.1.5 Can sealing tool
 - 8.1.6 Graduated cylinder
 - 8.1.7 25 cc VWR Petri for soil and miscellaneous samples
 - 8.1.8 250 mL plastic jar for filters, soil, and miscellaneous samples
 - 8.1.9 Hot plate
 - 8.1.10 Teflon beakers and lids
 - 8.1.11 1 L Marinelli beaker for soil samples
- 8.2 Instrumentation
 - 8.2.1 High purity germanium detector, with associated electronics and data reduction software



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8.2.2 Top loader balance

9.0 REAGENTS, CHEMICALS, AND STANDARDS

- 9.1 NIST traceable mixed gamma standard in 100 cc aluminum can
- 9.2 NIST traceable mixed gamma standard in 2.0 L Marinelli beaker
- 9.3 NIST traceable mixed gamma standard in 0.5 L Marinelli beaker
- 9.4 NIST traceable mixed gamma standard in Gelman Sciences 10 cc Petri dish
- 9.5 NIST traceable mixed gamma standard in 13, 47 mm glass fiber filter composites in Gelman Sciences Petri dish.
- 9.6 NIST traceable mixed gamma standard in 0.4 L jar
- 9.7 NIST traceable mixed gamma standard in 0.25 L jar
- 9.8 NIST traceable mixed gamma standard in 1, 47 mm glass fiber filter
- 9.9 NIST traceable mixed gamma standard in Impregnated Charcoal Sample Cartridge.
- 9.10 NIST traceable mixed gamma standard in VWR (53 mm x 15 mm) Petri dish (approximately 25 cc)
- 9.11 NIST traceable mixed gamma standard in aqueous solution
- 9.12 NIST traceable mixed gamma standard in 1.0 L Marinelli beaker
- 9.13 NIST traceable mixed gamma standard in 20 mL liquid scintillation vial
- 9.14 16 M Nitric acid, reagent grade (HNO₃)

10.0 SAMPLE HANDLING AND PRESERVATION

- 10.1 For soil samples, 500 g of sample should be collected, preferably in a plastic container to avoid breakage.
 - 10.2 For water samples, 2 L of sample should be collected in a plastic container and preserved to a pH < 2 with nitric acid.
 - 10.2.1 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should contact the Group Leader or Team Leader.
- NOTE:** If the analysis is requesting I-131 (or any other iodine isotopes) Analysis without preserving is acceptable. If a sample is preserved with acid without stabilizing the iodine, Iodine may volatilize and escape the solution as a gas.
- 10.2.2 If approved by the client, the analyst should adjust the pH with nitric acid to a pH < 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This



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acidification should be documented on a batch history sheet and attached to the batch paperwork.

10.3 For filters no preservation is necessary.

11.0 SAMPLE PREPARATION

11.1 Solid Sample Preparation.

11.1.1 Prepare the sample for gamma counting in accordance with SOP GL-RAD-A-021, Soil Sample Preparation for the Determination of Radionuclides.

11.1.2 Fill the appropriate container with sample prepared from step 11.1.1 using the following steps as a guideline:

11.1.2.1 If Ra-226 analysis is required, the sample is placed in a 100 cc can for in-growth.

NOTE: It is recommended that in-growth be allowed 20 days to quantify Ra-226. Shorter ingrowth periods can be used at the request of the client. However, shorter in-growth periods may decrease the accuracy of the data. If there is insufficient mass of sample to fill the 100 cc can, contact the Team or Group Leader.

11.1.2.2 If sufficient mass is available, homogenized samples should be placed in the 100 cc can. Determine the net weight of the sample. If the net weight is less than 54 g or greater than 192 g, contact the Team or Group Leader to determine the appropriate counting container. Record sample weight and date in AlphaLIMS and on sample container.

11.2 Water Sample Preparation

11.2.1 Place the appropriate labeled Marinelli beaker (typically 500 mL or 2 L) on a balance and tare the balance.

11.2.2 If less than approximately 1.1 L is available, sample should be poured into a 500 mL Marinelli beaker.

11.2.3 Transfer the appropriate volume to the tared container and record the volume of the sample on the Queue sheet.

NOTE: If there is insufficient sample to fill the Marinelli, record the exact amount of sample volume on the container and on the Queue sheet. Dilute the sample to the appropriate volume to maintain the calibration geometry. Record the volume the sample was diluted to on the sample container, also.

11.2.4 The MB should be recorded on the Queue sheet to be the same aliquot as the largest sample in the batch. An empty Marinelli beaker should be labeled as the MB and submitted with each batch of samples.



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11.2.5 Submit the Marinellis and completed paperwork to the count room for gamma counting analysis.

11.3 Urine Sample Preparation

11.3.1 Refer to GL-RAD-B-030.

11.4 Preparation of Miscellaneous Matrices

11.4.1 Prepare the sample in accordance with GL-RAD-A-026 for The Preparation of Special Matrices for the Determination of Radionuclides.

11.4.2 If sample(s) was (were) received from the client in a container that matches a calibrated geometry, a direct count of the sample can be performed.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

Refer to GL-RAD-D-003.

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

Refer to GL-RAD-I-001.

14.0 ANALYSIS AND INSTRUMENT OPERATION

Refer to GL-RAD-I-001.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-010.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation and reduction take place in accordance with SOP GL-RAD-D-003 and GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Data are reviewed and packaged in accordance with GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as Quality Documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

20.1 USEPA. Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Method 901.1, August 1980.

20.2 Canberra Nuclear Genie System Spectroscopy, Applications and Display User's Guide, Vol. I and II, May 1991.

20.3 DOE EML Procedures Manual, HASL-300, 27th Edition. (Section 4.5.2.3)



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20.4 DOE EML Procedures Manual, HASL-300, 28th Edition. (Ga-01-R)

20.5 Manual for the Certification of Laboratories Analyzing Drinking Water. Criteria and Procedures Quality Assurance. Fifth Edition EPA 815-R-05-004 January 2005.

21.0 HISTORY

Revision 22: Updated ingrowth period for Ra-226 to 20 days.

Revision 23: Procedure updated to include requirements for drinking water samples.

Revision 24: Changed recovery limit for laboratory fortified blank from 90-100% to 90-110% in section 3.3.

Revision 25: Type II to type I water.

Revision 26: Removed reference to obsolete software. Updated the reagents and standards section. Updated sample prep section to current practices.



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SOP Effective Date 12/08/92
Revision 16 Effective July 2016

Digestion for Soil

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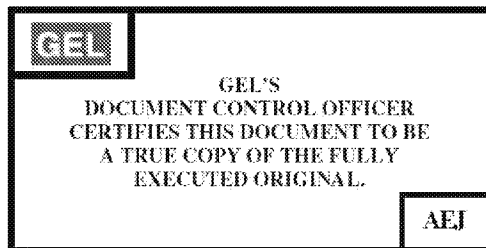
VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR DIGESTION FOR SOIL

(GL-RAD-A-015 REVISION 16)

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1.0 STANDARD OPERATING PROCEDURE FOR DIGESTION FOR SOIL**2.0 METHOD OBJECTIVE, PURPOSE, CODE, AND SUMMARY**

- 2.1 This standard operating procedure provides the necessary instructions to conduct digestion on soil type matrices. An appropriate amount of sample is aliquotted into either a teflon or a glass container. Concentrated acids and heat are applied until a homogenous aqueous solution is produced. This is accomplished by an acid dissolution or a fusion technique.
- 2.2 This procedure is applicable to soils for any analysis requiring complete dissolution. Sample aliquots of one gram or less can be accommodated by the equipment and amount of reagents specified. Sequential analysis of more than one radionuclide may be accomplished by the addition of carriers and tracers for each applicable analysis prior to digestion.
- 2.3 To minimize errors due to non-homogeneity of a sample, it is always desirable to use the largest possible aliquot of sample. The existence of "hot particles" may lead to questionable data when very small sample aliquots are used. In cases where radionuclide concentrations are "higher than normal environmental levels" and smaller aliquots are necessary, it is preferable to dilute the digested 1 gram sample rather than select a smaller aliquot. The analyst should consult the group leader for guidance in these situations.

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

Method Detection Limit (MDL): Refer to the specific analytical SOP for minimum detectable activity (MDA) values.

4.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a group leader or senior technical specialist. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

- 5.1 Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.
- 5.2 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.
- 5.3 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
- 5.4 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.



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- 5.5 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 5.6 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.
- 5.7 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.
- 5.8 Deionized (DI) water: Type I DI water, Refer to GL-LB-E-016.

6.0 INTERFERENCES

Not Applicable

7.0 SAFETY PRECAUTIONS AND WARNINGS

- 7.1 Personnel performing this analytical procedure are trained to the safe laboratory practices outlined in the Safety, Health, and Chemical Hygiene Plan, GL-LB-N-001.
- 7.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.
- 7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.
- 7.4 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a group or team leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

- 8.1 Equipment
- 8.1.1 Beakers (Glass and Teflon of various sizes)
 - 8.1.2 Platinum crucibles
 - 8.1.3 Hot plate
 - 8.1.4 Blast burner
 - 8.1.5 PTFE spatula
 - 8.1.6 High temperature resistant gloves
 - 8.1.7 Tongs
 - 8.1.8 Segmented muffle furnace
 - 8.1.9 Watch glasses (various sizes)
 - 8.1.10 Zirconium crucibles and lids
 - 8.1.11 Aluminum Foil



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- 8.1.12 Analytical balance
- 8.1.13 Polypropylene centrifuge tube (50 mL)

9.0 REAGENTS AND STANDARDS

9.1 Reagents

- 9.1.1 Concentrated Nitric Acid, reagent grade (16 M HNO_3)
- 9.1.2 Concentrated Hydrofluoric Acid, (49% HF).
- 9.1.3 Concentrated Hydrochloric Acid, reagent grade (12 M HCl)
- 9.1.4 Deionized (DI) Water
- 9.1.5 Saturated Boric Acid, 5%: Dissolve 50 g of H_3BO_3 per liter of DI water.
- 9.1.6 Ammonium Hydroxide (28-30% NH_4OH)
- 9.1.7 Potassium Fluoride, anhydrous (KF)
- 9.1.8 Sodium Sulfate (Na_2SO_4)
- 9.1.9 Concentrated Sulfuric Acid, (18 M H_2SO_4)
- 9.1.10 Sodium Hydroxide pellets
- 9.1.11 1.25 M Calcium Nitrate: Dissolve 205 g of anhydrous Calcium Nitrate or 295 g hydrated Calcium Nitrate, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, in 500 mL of DI water. Dilute to 1 L with DI water.
- 9.1.12 Iron Carrier (10 mg/mL): Dissolve 62.7 g of $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ or 72.3 g of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in 800 mL DI water and dilute to 1 L with DI water.

9.2 Standards

- 9.2.1 See applicable analytical SOP for the appropriate standards.

10.0 SAMPLE HANDLING AND PRESERVATION

Soil and sand require no preservation and may be shipped in any suitable container.

11.0 SAMPLE PREPARATION

- 11.1 For instructions on drying, grinding, homogenizing, and blending soil samples, refer to GL-RAD-A-021 for Soil Sample Preparation for the Determination of Radionuclides.
- 11.2 Alpha Spec soil digestion procedure (also to be used for total Uranium digestions).
 - 11.2.1 If samples are to be ashed, weigh an appropriate aliquot (0.1 to 1.0 g) into a glass container. If dried sample used, the sample aliquot should be placed in teflon beaker. If required, the DUP, MS, and MSD should be the same aliquot as the appropriate sample referenced on the Queue sheet. Record all aliquots on the Queue sheet. For alpha spec, add approximately 4 to 6 drops of Iron Carrier to the blank and LCS beakers.



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The blank and LCS aliquot should be recorded on the Queue sheet to be the same aliquot as the largest sample in the batch. Iron Carrier may also be added to all of the samples in the batch, if needed, based on the appearance and iron content of each sample. If Solid Reference Material is required, weigh out approximately 0.1 g into the LCS beaker, and record the exact weight on the Queue sheet.

11.2.2 Add appropriate spikes and tracers according to the analytical SOP you are performing. Record tracer and spike IDs and volumes on Queue sheet. If dried samples used, proceed to step 11.2.5.

11.2.3 Place each glass container in the furnace with even spacing. A removable shelf is provided that can be used if necessary. Cover glass containers prior to closing door to furnace to prevent cross-contamination. Cover with a material appropriate for the sample, such as aluminum foil or glass. If aluminum foil is used, poke holes in the foil so that pressure does not build up inside the container. Muffle for approximately 2 to 4 hours. Use tongs and/or wear protective gloves to remove the containers from the furnace when the furnace temperature has decreased below 100° C.

NOTE: Determining ashing routine is based on analyst experience with the matrix. If flammability of the matrix is in question, such as vegetation, tissues, and paper, use the slow ramp procedure. (Consult operation method to set up procedure.) The time required to obtain an ash sample will vary depending on the type of material, size of sample, oven type, and capacity, and other factors. The influence of these factors generally can be established by good judgment, and experience with the materials being tested and the apparatus being used.

11.2.4 Add 5 to 10 mL concentrated Nitric Acid and 5 to 10 mL concentrated Hydrochloric Acid to each container and cover with a watch glass. Reflux for approximately 30 minutes. Transfer each sample into a previously labeled, clean teflon beaker. Rinse any remaining soil from the glass container into the teflon beaker using approximately 5 to 10 mL of concentrated Hydrochloric Acid. Dry on a hot plate using medium heat.

NOTE: All glass containers should be disposed of after muffling.

11.2.5 Add 10 mL of 49% Hydrofluoric Acid to each teflon beaker. Cover beakers with a teflon cover and place on hot plate on medium heat for approximately 30 minutes. Remove lids and evaporate solution to dryness on medium heat.

11.2.6 Add 10 mL of concentrated Nitric Acid and 10 mL of 49% Hydrofluoric Acid to each teflon beaker. Cover beakers with a teflon cover and place



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- on medium heat for approximately 30 minutes. Remove lids and evaporate the solution to dryness on medium heat.
- 11.2.7 Add 10 mL of concentrated Hydrochloric Acid and 5 mL of 49% Hydrofluoric Acid to each teflon beaker. Cover beakers with a teflon cover and place on medium heat for approximately 30 minutes. Remove lids and evaporate the solution to dryness on medium heat.
- 11.2.8 Add 10 mL concentrated Hydrochloric Acid and 10 mL concentrated Nitric Acid to each teflon beaker and evaporate to dryness on medium heat.
- 11.2.9 Add 10 mL concentrated Hydrochloric Acid and 1 mL of saturated Boric Acid to each sample and evaporate to dryness on medium heat.
- 11.2.10 Add 5 mL concentrated Nitric Acid and evaporate to dryness on medium heat (Th, U by ICPMS and Ra samples only).
- 11.2.11 Digestion should be complete at this point. Proceed to appropriate analytical procedure for analysis. For alpha spec analyses, if further clean up of sample is deemed necessary by your team leader/group leader, continue with step 11.2.12.
- 11.2.12 Dissolve the sample residue in 10 to 15 mL of concentrated Hydrochloric Acid and transfer to a disposable centrifuge tube using DI water as a rinse. Add Ammonium Hydroxide until Iron Hydroxide precipitates, then add 2 mL excess Ammonium Hydroxide. Centrifuge sample and discard supernate.
- 11.2.13 Wash precipitate with 20 mL of DI water that has been adjusted to pH 10 with Ammonium Hydroxide. Centrifuge sample and discard supernate.
- 11.2.14 For any combination of alpha spec analyses, dissolve the Iron precipitate in the required reagent listed in the appropriate analytical procedure being used and proceed per SOP.
- 11.3 Gross Alpha/Beta Soil Digest Procedure
- 11.3.1 Weigh out an appropriate aliquot (normally 0.1 g) into a teflon beaker. If required, the DUP, MS, and MSD should be the same aliquot as the appropriate sample referenced on the Queue sheet. Record all aliquots.
- 11.3.2 Add spike solutions to the applicable samples and record volumes.
- 11.3.3 Add 10 mL of concentrated Nitric Acid to each sample.
- 11.3.4 Place samples on medium heat and cover each sample with a teflon lid. Reflux all samples for approximately 30 minutes.



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- 11.3.5 Remove teflon lids and add 5 mL concentrated Hydrochloric Acid and 10 mL 49% Hydrofluoric Acid to each sample. Cover samples and reflux for approximately 120 minutes.
- 11.3.6 Remove teflon lids and allow samples to evaporate to dryness on medium heat.
- 11.3.7 Add 5 mL of concentrated Nitric Acid and evaporate to dryness on medium heat.
- 11.3.8 Repeat Step 11.3.7.
- 11.3.9 Add 5 mL of concentrated Nitric Acid to the dry samples. Place the samples back on the hot plate long enough so that the dried sample dissolves into the acid.
- 11.3.10 Samples are now ready for planchetting in accordance with GL-RAD-A-001B.

11.4 Pyrosulfate Fusion Dissolution Procedure

NOTE: Leather gloves and a leather apron should be worn when working in the fusion hood. All equipment should be checked prior to fusing samples. Extreme caution should be taken when working with open flames.

- 11.4.1 Add appropriate tracers to each sample as well as to control samples according to the analytical SOP you are performing. Prepare the laboratory control samples and spikes in the same manner as the other samples in the batch.
- 11.4.2 Transfer the dried, tared sample into a Platinum dish. Wet the sample with approximately 1 mL concentrated Nitric Acid in order to eliminate spattering from Carbonates.
- 11.4.3 When the bubbling has ceased, add about 1 mL 49% Hydrofluoric Acid and heat to dryness on a medium hot plate.
- 11.4.4 Once the sample is dry, add approximately 3 g of Potassium Fluoride; using a PTFE spatula, stir the dried sample and Potassium Fluoride together. Fuse the sample over a blast burner until all bubbling has ceased, sample has visibly dissolved, and clear fusion flux is observed.
- 11.4.5 Remove from the burner, place on a hot plate using medium heat, and let equilibrate to the hot plate temperature.
- 11.4.6 Slowly add about 4 mL concentrated Sulfuric Acid rinsing down the walls of the Platinum dish to the extent possible. Let the sample set on the hot plate until bubbling has ceased.
- 11.4.7 Add approximately 2 g of Sodium Sulfate to the sample. Heat over the blast burner again in order to transpose the Fluoride cake into a



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Pyrosulfate cake. Heat until bubbling ceases and most of the liquid is gone. Place on the hot plate to cool to the hot plate's temperature.

11.4.8 Add approximately 5 mL concentrated Hydrochloric Acid and approximately 35 mL DI water to the fusion cake while heating to effect dissolution. Take volume up to about 100 mL with DI water.

11.4.9 Proceed to the applicable SOP for analysis.

11.5 Sodium Hydroxide Fusion Dissolution Procedure

11.5.1 Add appropriate tracers to each sample as well as to control samples according to the analytical SOP you are performing. Prepare the laboratory control samples and spikes in the same manner as the other samples in the batch.

NOTE: For Ra analyses, if an empty crucible is analyzed as a reagent blank, 100 mg Ca (by evaporating 2 mL of 1.25 M Calcium Nitrate) should be added to the Zirconium crucible.

11.5.2 Place crucibles on a hot plate and heat to dryness on medium heat.

11.5.3 Remove crucibles from hot plate and allow to cool.

11.5.4 Add 10-15 grams of Sodium Hydroxide to the crucibles.

11.5.5 Place the crucibles with lids in the 600 °C furnace using tongs.

11.5.6 Fuse crucibles for 15-20 minutes.

NOTE: Longer times may be needed for larger particle sizes.

11.5.7 Remove hot crucibles from furnace very carefully using tongs, and transfer to hood after crucibles have cooled.

11.5.8 Add approximately 25-50 mL of water to each and heat on hotplate to loosen/dissolve solids.

11.5.9 If necessary for dissolution, add more water and warm as needed on a hotplate.

11.5.10 Proceed to applicable SOP for analysis.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

12.1 Method Verification

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Control Requirements

See specific SOP for specific method quality control requirements.

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

Refer to GL-LB-E-002 and GL-LB-E-004.

14.0 ANALYSIS AND INSTRUMENT OPERATION



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Refer to GL-LB-E-002 for Balances.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-LB-E-002 for Balances.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Refer to GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Not Applicable

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material are be handled and disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

None

21.0 HISTORY

Revision 13: Technical: Updated section 11.2.10. Updated Appendix I to reflect change to section 11.2.10.

Revision 14: Updated sections 8.0 and 9.0. Added Section 11.5.

Revision 15: Technical updates for SOP consistency as part of annual review.

Revision 16: Updated Sample preparation section with current volume of Acid used in process



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APPENDIX 1

ALPHA SPECTROSCOPY SOIL DIGESTION

- _____ Weigh appropriate amount of dry soil to meet MDA
- _____ Add appropriate tracers and spikes to samples
- _____ Muffle samples for approximately 2-4 hours
- _____ Add ~10 mL concentrated HNO_3 and ~10 concentrated HCl
- _____ Reflux for approximately 30 minutes
- _____ Transfer to teflon
- _____ Evaporate to dryness on medium heat
- _____ Add 10 mL concentrated HF
- _____ Reflux for approximately 30 minutes
- _____ Evaporate to dryness on medium heat
- _____ 10 mL concentrated HNO_3 and 10 mL concentrated HF
- _____ Reflux for approximately 30 minutes
- _____ Evaporate to dryness on medium heat
- _____ 10 mL concentrated HCl and 5 mL concentrated HF
- _____ Reflux for approximately 30 minutes
- _____ Evaporate to dryness on medium heat
- _____ 10 mL concentrated HNO_3 and 10 mL concentrated HCl
- _____ Evaporate to dryness on medium heat
- _____ 10 mL concentrated HCl and 1 mL boric acid
- _____ Evaporate to dryness on medium heat
- _____ For Thorium, Uranium by ICPMS or Radium samples add 5 mL concentrated HNO_3 and evaporate to dryness on medium heat
- _____ Proceed to the appropriate procedure for completion of analysis



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APPENDIX 2

GROSS ALPHA/BETA SOIL DIGESTION

- _____ Weigh appropriate amount of dry soil to meet MDA
- _____ Add appropriate spikes to samples
- _____ 10 mL concentrated HNO_3
- _____ Reflux for approximately 30 minutes
- _____ 5 mL concentrated HCl and 10 mL concentrated HF
- _____ Reflux for approximately 120 minutes
- _____ Evaporate to dryness on medium heat
- _____ 5 mL concentrated HNO_3
- _____ Evaporate to dryness on medium heat
- _____ 5 mL concentrated HNO_3
- _____ Evaporate to dryness on medium heat
- _____ Dissolve sample with 5 mL concentrated HNO_3
- _____ Proceed to GL-RAD-A-001B for completion of analysis



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VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE
FOR
THE ISOTOPIC DETERMINATION
OF THORIUM

(GL-RAD-A-038 REVISION 17)

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1.0 STANDARD OPERATING PROCEDURE FOR THE ISOTOPIC DETERMINATION OF THORIUM

2.0 METHOD OBJECTIVE, PURPOSE, AND SUMMARY

- 2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic thorium in a variety of matrices.
- 2.2 A soil/solid sample is aliquoted and digested, if necessary. The elements are then separated through ion exchange resins. For liquid samples, actinide elements are scavenged by coprecipitation with iron hydroxide. The precipitate is dissolved, and separation of elements is accomplished through ion exchange resins. The elements are then prepared for the measurement of radioactive isotopes by coprecipitation with neodymium fluoride. The neodymium fluoride precipitate is trapped on a filter, mounted on a metal disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.
- 2.3 GEL utilizes methods that are derived from established sources. This method is based on the source method from DOE EML Methods Manual HASL 300 PU-02, 03 and uses similar principles of radiochemical separation and counting. This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800: "Sequential Separation of Americium and Plutonium by Extraction Chromatography." This method is also based on the source method EPA 053917 EMSL LV 1979 "Isotopic Determination of Plutonium, Uranium, and Thorium in Water, Soil, Air, and Biological Tissue" and in some cases this method is referenced as EPA RA-LV-PI.
- 2.4 This method has been modified on the basis of GEL's Performance Based Measurement System (PBMS).

3.0 METHOD APPLICABILITY

- 3.1 Method Detection Limit (MDL): Typical minimum detectable activity (MDA) for samples analyzed for thorium is 1 pCi/L or 1 pCi/g for all isotopes.
- 3.2 Analyst training records are maintained as quality records outlined in GL-QS-E-008. Analyst training and proficiency in the method is outlined in GL-QS-E-011 for Method Validation and Initial and Continuing Demonstrations of Capability.
- 3.3 Applicable matrices to this SOP are liquids, drinking water, vegetation, tissues, air filters, and solids.

NOTE: This method is not an EPA approved method for the analysis of drinking water.

4.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group or Team Leader. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

- 5.1 AlphaLIMS: GEL's Laboratory Information Management System.

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- 5.2 Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.
- 5.3 Deionized (DI) water: Type I water, Refer to GL-LB-E-016.
- 5.4 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.
- 5.5 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
- 5.6 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.
- 5.7 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 5.8 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.
- 5.9 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.
- 5.10 Solid Reference Material (SRM): A solid material containing known and verified amounts of analytes.
- 5.11 Tracer: A known quantity of a radioisotope that is added to each sample of a chemically equivalent radioisotope of unknown concentration so that the yield of the chemical separation can be calculated.

6.0 INTERFERENCES

- 6.1 Internal tracer standards may have ingrown daughters that may interfere with the analysis. For example, Th-228 will be present in aged U-232 standard. This problem is overcome by not performing thorium and uranium analysis in tandem.
- 6.2 Short-lived radioactive progeny may ingrow on prepared filters. For example, the Ra-224 alpha peak will be present if the Th-228 parent is present. These interferences are minimized by counting samples as soon as possible after separation chemistry.

7.0 SAFETY PRECAUTIONS AND WARNINGS

- 7.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.
- 7.2 Personnel handling radioactive materials are trained in and follow the procedures

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outlined in GL-RAD-S-004 for Radioactive Material Handling.

- 7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.
- 7.4 If there is any Question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

8.1 Apparatus and Equipment

- 8.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities
- 8.1.2 Vacuum pump and filtration rig
- 8.1.3 Disposable filter funnels (containing 25 mm filters with 0.1 μm pore size)
- 8.1.4 Metal disks, 29 mm diameter
- 8.1.5 Stainless steel tweezers
- 8.1.6 Polypropylene centrifuge tube (50 mL)
- 8.1.7 Sample drying and ashing apparatus
- 8.1.8 Sample homogenization apparatus
- 8.1.9 Analytical Grade 1X8 anion exchange resin, 100-200 mesh
- 8.1.10 Beakers (Glass and Teflon of various sizes)
- 8.1.11 2.5 cm^3 column
- 8.1.12 25 mL column funnel extension
- 8.1.13 Watch glasses (various sizes)
- 8.1.14 Hot plate
- 8.1.15 Pipettes
- 8.1.16 Balances
- 8.1.17 Disposable transfer pipettes
- 8.1.18 pH strips

9.0 REAGENTS AND STANDARDS

9.1 Reagents

- 9.1.1 Hydrogen Peroxide (30% H_2O_2)
- 9.1.2 Neodymium (500 mg/L Nd)
- 9.1.3 Neodymium (10,000 mg/L Nd)
- 9.1.4 Ethyl alcohol (80% EtOH): Dilute 400 mL ethanol to 500 mL with DI water.
- 9.1.5 Hydrochloric Acid, concentrated (12 M HCl)
- 9.1.6 Hydrofluoric Acid, concentrated (49% HF)

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- 9.1.7 Iron Carrier (10 mg/mL): Dissolve 62.7 g of $\text{Fe}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ or 72.3 g $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in 800 mL DI water and dilute to 1 L with DI water.
- 9.1.8 Hydrochloric Acid (9 M HCl): Add 750 mL of concentrated hydrochloric acid to 100 mL of DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.9 Hydrochloric Acid (2 M HCl): Add 167 mL of concentrated hydrochloric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.
- 9.1.10 Nitric Acid, concentrated (16 M HNO_3)
- 9.1.11 Nitric Acid (8 M HNO_3): Add 500 mL concentrated nitric acid to 500 mL DI water.
- 9.1.12 Cellulose Nitrate Membrane filters (47 mm)
- 9.1.13 Substrate Suspension: Dilute 4 mL of neodymium (10,000 mg/L) and 80 mL concentrated hydrochloric acid to 1500 mL with DI water. Add, while stirring, 40 mL of 49% hydrofluoric acid and dilute to 2 L with DI water.
- 9.1.14 Sulfuric Acid, concentrated (36 M H_2SO_4)
- 9.1.15 Ammonium Hydroxide, concentrated (28-30% NH_4OH)
- 9.1.16 Saturated Boric Acid, 5%: Dissolve 50 g of H_3BO_3 per liter of DI water.
- 9.2 Standards
 - 9.2.1 NIST traceable standards: Th-229, Th-230, Th-232, Ac-227.
 - 9.2.2 Refer to GL-RAD-M-001.

10.0 SAMPLE HANDLING AND PRESERVATION

- 10.1 Liquid samples should be preserved to $\text{pH} < 2$ with concentrated nitric acid and collected in a plastic bottle.
- 10.2 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a $\text{pH} > 2$, the analyst should contact the Group Leader or Team Leader. If approved by the client, the analyst should adjust the pH with concentrated nitric acid to a $\text{pH} < 2$. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This acidification should be documented on a batch history sheet and attached to the batch paperwork.
- 10.3 If the sample has exceeded the hold time, the analyst should contact the Group Leader before continuing with the batch.
- 10.4 Soil samples require no preservation and may be shipped in any suitable container.

11.0 SAMPLE PREPARATION

NOTE: Aliquots may be estimated by using the count time estimator.

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11.1 Soil/Solids Sample Preparation

11.1.1 If not already done, dry and homogenize the sample by performing GL-RAD-A-021.

11.1.2 Measure an appropriate aliquot of soil/solids (usually 0.2 g to 1.0 g) in a suitable container (glass or Teflon beaker). If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced on the sheet. Record all aliquots on the Queue sheet. Add approximately 4 to 6 drops of iron carrier to the MB and LCS beakers. The MB and LCS aliquots should be recorded on the Queue sheet to be the same aliquot as the largest sample in the batch. Iron carrier may also be added to all samples in the batch, if necessary, based on the appearance and iron content of each sample. If solid reference material is required, weigh out approximately 0.1 g into the LCS beaker and record the exact weight on the Queue sheet.

11.1.3 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference the batch Queue sheet and Pull sheet for client requirements to determine appropriate tracer and spike.

11.1.3.1 For the determination of isotopic thorium, Th-229 is typically used as the tracer, and Th-232 is typically used as the spike.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing on the Queue sheet.

11.1.4 If samples are to be placed in a muffle furnace (recommended for miscellaneous solid samples), samples shall be in a glass beaker (not Teflon). Refer to GL-RAD-A021B for instructions.

11.1.5 For thorium analysis, digest as specified in GL-RAD-A-015.

11.1.6 Proceed to step 11.2.6.

11.2 Aqueous Sample Preparation

11.2.1 Add an appropriate aliquot of sample to a labeled beaker. Prepare a MB and LCS using DI water and a small amount of concentrated nitric acid to a pH < 2. The volume of DI water used should be the same as the largest volume of sample in the batch. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced on the Queue sheet. Record all aliquots on the Queue sheets.

11.2.2 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference the batch Queue sheet and Pull sheet for client

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requirements to determine appropriate tracer and spike.

11.2.2.1 For the determination of isotopic thorium, Th-229 is typically used as the tracer, and Th-232 is typically used as the spike.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing on the Queue sheet.

NOTE: Other sample matrices, such as vegetation, air filters, tissue, etc., are prepared as outlined in GL-RAD-A-026. The analyst must ensure that the appropriate tracer(s) is added to these other matrices as discussed in section 11.1.3 and 11.2.2.

NOTE: If samples contain large amounts of sediment and client requires inclusion with the liquid portion of the sample, proceed to section 11.3.

11.2.3 Add 1 mL of iron carrier (10 mg/mL).

11.2.4 Add concentrated ammonium hydroxide until turbidity persists, or the pH > 9. Add approximately 2 mL in excess. Heat to boiling for approximately 10 minutes or until precipitate breaks into fine particles. Allow to settle and cool.

11.2.5 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard supernate.

NOTE: Exercise care in this step because finely divided material that contains the actinides may also be present in addition to large iron hydroxide flocks.

11.2.6 Dissolve the precipitate from step 11.2.5 or residue from step 11.1.6 in 10 to 15 mL of 8 M nitric acid.

NOTE: If all of the precipitated iron solids do not go into solution, it may be necessary to place samples in a hot water bath for approximately 10-15 minutes before proceeding to column work.

11.2.7 Slurry Analytical Grade 1X8 anion exchange resin in DI water. Transfer the resin to a small column to obtain a settled resin bed of approximately 2.5 mL.

11.2.8 Condition the column with 15 mL of 8 M nitric acid.

11.2.9 Pass the solution from step 11.2.6 through the column, and collect in a drip pan for disposal.

NOTE: If the samples were traced with Ac-227, annotate the separation date and time when the load solution has passed through the column.

11.2.10 Rinse the column with 5 mL of 8 M nitric acid, and collect in a drip pan for disposal.

11.2.11 Rinse the column with 15 mL of 8 M nitric acid, and collect in a drip

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pan for disposal.

- 11.2.12 Elute the thorium from the column by adding 20 mL of 9 M hydrochloric acid, collecting the elution in a labeled, disposable centrifuge tube.
- 11.2.13 Transfer the sample to a beaker using DI water. Add approximately 4 to 6 drops of iron carrier. Evaporate the sample to dryness on medium heat.
- 11.2.14 Add 10 mL of concentrated nitric acid and slowly add approximately 2 to 3 mL hydrogen peroxide. Evaporate samples to dryness on medium heat.
- 11.2.15 Add 2 mL of concentrated hydrochloric acid and evaporate samples to dryness on medium heat.
- 11.2.16 Dissolve the residue with 4 mL of 2 M hydrochloric acid and transfer the sample to a clean centrifuge tube using DI water.
- 11.2.17 Add 0.1 mL of 500 mg/L neodymium and swirl. Add 2.0 mL of 49% hydrofluoric acid and swirl. Wait approximately 30 minutes to allow fluorides to coprecipitate with thorium.
- 11.2.18 Place the disposable filter funnel on the filter rig and apply vacuum.
- 11.2.19 Rinse the funnel with 80% ethyl alcohol.
- 11.2.20 Add 5 mL substrate suspension. After solution has passed through filter, add another 5 mL of substrate suspension.
- 11.2.21 Filter the fluoride precipitated solution through the filter paper. Rinse the centrifuge tube with approximately 5 mL DI water and pass through filter.
- 11.2.22 Rinse the funnel with 80% ethyl alcohol.

CAUTION: Directing a stream of liquid onto the filter will disturb the distribution of the precipitate on the filter and render the sample unsuitable for alpha spectrometry resolution.

- 11.2.23 Without turning off the vacuum, remove the funnel.
- 11.2.24 Turn off vacuum and remove filter. Mount filter on a labeled 29 mm flat planchet. Ensure that the filter is centered and as flat as possible on the planchet.

NOTE: Care should be taken not to touch the active area of the filter with tweezers.

- 11.2.25 Place the mounted filter under a heated lamp to dry (usually for 10-20 minutes).
- 11.2.26 Submit samples for alpha spectrometer counting.

11.3 Samples Containing Large Amounts of Sediment:

NOTE: When aliquoting samples that contain large amounts of sediment, ensure sample is thoroughly homogenized.

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11.3.1 Dry on medium to low heat.

11.3.2 Muffle in a furnace at approximately 550° C for a minimum of 2 hours.

11.3.3 Proceed to step 11.1.5.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

NOTE: Client contractual QC requirements override the requirements in this section.

12.1 Method Verification Requirements

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Requirements

12.2.1 A Method Blank (MB) should accompany each batch of 20 or less samples. The reported value of the MB should be less than or equal to the Contract Required Detection Limit (CRDL).

12.2.2 The tracer added to all samples is used to calculate the method recovery. The method recovery of all samples should be between 15-125% when compared to the reference standard.

12.2.3 A duplicate (DUP) sample should be run with each batch of 20 or less samples. The relative percent difference (RPD) between the actual sample and the DUP should be less than or equal to 20% if both the sample and DUP results are greater than 5 times the minimal detectable concentration (MDC), or 100% if they are both less than 5 times MDC. If both results are less than the MDC, then limits are not applicable.

12.2.4 A Laboratory Control Sample (LCS) should be run with each batch of 20 or less samples. The recovery of the LCS should fall between 75-125%.

12.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the QC criteria from 12.2.1 through 12.2.4 cannot be satisfied, the analyst should inform the Group Leader and initiate a Data Exception Report as outlined in GL-QS-E-004.

13.0 INSTRUMENT CALIBRATION AND PERFORMANCE

For direction on calibration and instrument performance, refer to GL-RAD-I-009.

14.0 ANALYSIS AND INSTRUMENT OPERATION

For analysis and instrument operation, refer to GL-RAD-I-009.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

For maintenance of system, refer to GL-RAD-I-010.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation, and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Refer to GL-RAD-D-003 for instructions concerning the data review process, approval, and transmittal.

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18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material are disposed as outlined in the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

- 20.1 EPA Environmental Monitoring and Support Laboratory, Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.
- 20.2 EML Procedures Manual HASL-300, 1982.
- 20.3 Analytical Chemistry. Rapid Determination of Th-230 in Mill Tailings by Alpha Spectroscopy. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol. 59, No. 21, 1987.
- 20.4 Los Alamos Health and Environmental Chemistry: Analytical Techniques. LA-10300-M Vol. 1, September 1987.
- 20.5 Special thanks to Dr. Bill Burnett and his associates for assistance in developing this method at Florida State University.
- 20.6 U.S. DOE RP 800., Methods for Evaluating Environmental and Waste Management Samples, "Sequential Separation of Americium and Plutonium by Extraction Chromatography", 1997.
- 20.7 U.S. EPA, EMSL LV 053917 (EPA RA-LV-PI), "Isotopic Determination of Plutonium, Uranium, and Thorium in Water, Soil, Air, and Biological Tissue", 1979.

21.0 HISTORY

Revision 13: Changed stainless steel to metal in sections 2.2 and 8.1.4.

Revision 14: Added section 2.3 and references for certification.

Revision 15: Updated reference to calculation SOP and DI water.

Revision 16: Technical updates for SOP consistency as part of annual review. Added Appendix 3 regarding clean up steps.

Revisions 17: Updated SOP to current practices.

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APPENDIX 1

THORIUM

Use a 2.5 cm³ column with 1 x 8 anion resin (Cl⁻ form 100-200 mesh)

- _____ 15 mL 8 M HNO₃ (Condition Column)
- _____ 10–15 mL 8 M HNO₃ (Load)
- _____ 5 mL 8 M HNO₃ (Rinse)
- _____ 15 mL 8 M HNO₃ (Rinse)
- _____ **Elute Th:** 20 mL 9 M HCl (Catch in C-Tube)
- _____ Transfer to a clean beaker. Add approximately 4-6 drops of Fe carrier and evaporate to dryness on medium heat
- _____ 10 mL of concentrated HNO₃ and approximately 2–3 mL H₂O₂. Evaporate to dryness on medium heat.
- _____ 2 mL of concentrated HCl and evaporate to dryness on medium heat.
- _____ Dissolve with 4 mL of 2 M HCl and transfer to centrifuge tube with DI water.
- _____ Proceed to Appendix 2 for thorium precipitation

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APPENDIX 2

THORIUM PRECIPITATION

- _____ 0.1 mL 500mg/L neodymium and swirl
- _____ 2.0 mL 49% HF and swirl
- _____ Wait approximately 30 minutes
- _____ Filter



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APPENDIX 3

SAMPLE CLEANUP FROM AN ALPHA SPEC FILTER

1. Remove the filter from the mounting disc by wetting the filter with a small amount of acetone and pulling the filter off the disk using tweezers. Place the filter in a labeled glass beaker.
2. Add 4-6 drops of iron carrier (10 mg/L), 10 mL of concentrated hydrochloric acid and 1.0 mL of 5% boric acid solution.
3. Fill the bulb end of a disposable pipette with DI water and turn upside down. Place on the filter in the glass beaker to ensure the filter remains submerged.
4. Heat on a hot plate for 30 minutes frequently stirring the filter, flipping it over then back, etc. Use the disposable pipette to stir and flip.
5. Remove the filter from the solution and rinse with 9M HCl. Do this over the glass beaker so the 9M HCl rinse falls back into the beaker.
6. Evaporate to dryness.
7. Convert with concentrated Nitric Acid and evaporate to dryness.
8. Proceed to section 11.2.6 and perform separations as specified.

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Gamma Spectroscopy System Operation

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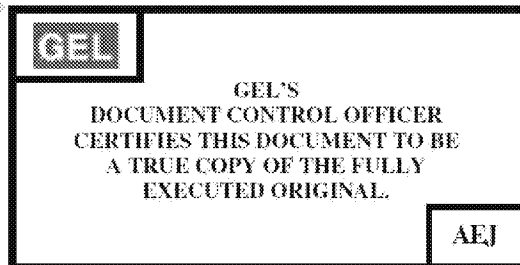
VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

**STANDARD OPERATING PROCEDURE
FOR
GAMMA SPECTROSCOPY SYSTEM OPERATION**

(GL-RAD-I-001 REVISION 21)

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1.0 STANDARD OPERATING PROCEDURE FOR GAMMA SPECTROSCOPY SYSTEM OPERATION**2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY**

- 2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for gamma isotopes using the Gamma Spectroscopy System.
- 2.2 Gamma emitting isotopes within the sample matrix are identified and quantified using gamma spectrometry. A sample aliquot is placed in a calibrated geometry and placed in the detector chamber. The germanium crystal therein produces a corresponding electrical pulse for the gamma photons that interact with the detector. The cumulative pulses are analyzed using software capable of quantifying gamma-emitting isotopes from the spectral data.

3.0 APPLICABLE MATRIX OR MATRICES

This is a nondestructive test for the measurement of gamma emitting isotopes in all matrices for which there is an available calibration standard.

4.0 METHOD SCOPE, APPLICABILITY AND DETECTION LIMIT

- 4.1 The aliquoted sample activity or sample position should be adjusted so that the detector system dead time remains less than 15%.
- 4.2 Method Detectable Activity: The MDA is based upon sample volume, instrument background, detector efficiency, count time and other statistical factors, as well as specific isotopic values such as abundance and half-life.

5.0 METHOD VARIATIONS

Not applicable

6.0 DEFINITIONS

- 6.1 Abundance: The combination of the isotopic decay branching ratio and the expected gamma emissions per disintegration of an isotope at a particular energy.
- 6.2 Key Line: The line chosen by the builder of the library to be the prominent line of the isotope. This line is used for the purposes of calculating activity, error and MDA.
- 6.3 AlphaLIMS: The Laboratory Information Management System used to store and report data.
- 6.4 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.
- 6.5 Refer to GL-QS-B-001 the Quality Assurance Plan for additional Lab-wide used definition.

7.0 INTERFERENCES/LIMITATIONS

- 7.1 Some gamma isotopes emit gamma lines that may overlap with those from other isotopes. If the energies of the two isotopes are within the energy tolerance setting, the peaks may not be resolvable and may give a positive bias to the result. This problem is minimized by careful review of the peak search.



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8.0 SAFETY PRECAUTIONS AND WARNINGS

Follow safety precautions as outlined in GL-LB-N-001 for the Safety, Health and Chemical Hygiene Plan.

9.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION**9.1 Apparatus and Equipment**

- 9.1.1 Compaq/DEC Alpha Station with OpenVMS
- 9.1.2 Canberra Genie-ESP Application Software
- 9.1.3 High purity germanium detector
- 9.1.4 Pulse processing electronics

10.0 REAGENTS AND STANDARDS**10.1 Standards**

- 10.1.1 NIST traceable mixed gamma standards in geometries and densities, closely approximating analytical samples, used to calibrate the instrument.

11.0 SAMPLE HANDLING AND PRESERVATION

Refer to GL-RAD-A-013 The Determination of Gamma Isotopes.

12.0 SAMPLE PREPARATION

Refer to GL-RAD-A-013 The Determination of Gamma Isotopes.

13.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

Refer to GL-RAD-A-013 The Determination of Gamma Isotopes.

14.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE**14.1 Calibration Standard**

- 14.1.1 Mixed Gamma calibrations typically use a standard with 8-12 photons emitted over a range from approximately 45 keV to approximately 2000 keV.
- 14.1.2 Single nuclide calibrations typically use a standard comprised of the nuclide of interest.

14.2 Verification Standard

- 14.2.1 Mixed Gamma calibrations- A second source (from different manufacturer or if from the same manufacturer, a different lot number) is used for verification. The lines from Am-241, Cs-137 and Co-60 are used to verify the efficiency curve. These encompass the low, middle and high portions of the energy range.
- 14.2.2 Single nuclide calibrations – A second source (from a different manufacturer or if from the same manufacturer, a different lot number) is used for verification.

14.3 Standardization

- 14.3.1 High Voltage Adjust



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14.3.1.1 See appropriate instrument manual for operation of electronics.

14.4 Calibration – Energy and efficiency calibrations are performed annually, upon initial instrument setup, after major repair or service, or when performance checks indicate a need.

NOTE: Expiration dates will match the last day of the month in which the calibration data was acquired.

14.4.1 Count Calibration Spectrum

14.4.1.1 Place the radioactive source on the detector.

14.4.1.2 Select **Calibration | Count a Calibration Standard** from the *Calibration* menu and click **OK**.

14.4.1.3 Enter the **Preset Live (secs):** in seconds and click **OK**. Count the standard until a minimum of 10,000 counts is acquired in each peak of interest.

14.4.2 Initial Energy & Shape Calibration

14.4.2.1 Select **Calibration | Initial Energy & Shape Calibration** from the *Calibration* menu and click **OK**.

14.4.2.2 Select the detector.

14.4.2.3 Select the **Certificate File** from the drop down list. Click **OK**.

14.4.2.4 From the *Energy Calibration* dialog box highlight one of the energy lines listed.

14.4.2.5 Move the cursor in the MCA window to the corresponding channel expected for that energy line.

14.4.2.5.1 The apex of the peak of interest should be at the expected channel.

14.4.2.5.2 From the *Energy Calibration* dialog box click the **Cursor** button.

14.4.2.6 Repeat the previous step until all energy lines listed have been referenced with a corresponding channel.

14.4.2.7 From the *Energy Calibration* dialog box select the **OK** button.

14.4.2.8 The system will ask “Do you want to do a full energy and shape calibration?” Select **YES**.

14.4.2.9 The energy and shape calibrations will now be performed with all of the lines from step 14.4.2.6. Verify the energy and shape curve generated. Select **OK** to continue or **Cancel** to abort the calibration.



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- 14.4.2.10 A new page will appear with the Energy Calibration Report and the FWHM Calibration Report. Review the columns marked difference. For the energy calibration, the absolute value of the difference must be less than 1.0 and for the FWHM calibration, the absolute value of the difference must be less than 0.5. Regardless of the results, select **Dismiss**.
- 14.4.2.11 A new pop-up screen will appear. If the results from the previous step were less than a 0.2 keV difference, select **OK**. If the results were greater than a 0.2 keV difference select **Cancel** and begin the energy calibration process again at step 14.4.1.
- 14.4.3 Energy Re-Calibrate
- 14.4.3.1 Select **Calibrate | Re-Calibrate | Energy and Shape Calibration** from the main menu and click **OK**.
- 14.4.3.2 Select the detector.
- 14.4.3.3 Select the certificate file and select the **OK** button.
- 14.4.3.4 The energy and shape calibrations will now be performed with all of the lines from step 14.4.2.6. Verify the energy and shape curve generated. Select **OK** to continue or **Cancel** to abort the calibration.
- 14.4.3.5 A new page will appear with the Energy Calibration Report and the FWHM Calibration Report. Review the columns marked difference. For the energy calibration, the absolute value of the difference must be less than 1.0 and for the FWHM calibration, the absolute value of the difference must be less than 0.5. Regardless of the results, select **Dismiss**.
- 14.4.3.6 A new pop-up screen will appear. If the results from the previous step were less than a 0.2 keV difference, select **OK**. If the were greater than a 0.2 keV difference select **Cancel** and begin the energy calibration process again. If it fails after re-calibration contact Group or Team Leader for further instructions.
- 14.4.4 Efficiency Calibrate
- 14.4.4.1 Select **Calibrate | Efficiency Calibrate** from the main menu.
- 14.4.4.2 Select the geometry that represents the standardized radioactive source and click **OK**. If the geometry doesn't exist select **Create New Geometry**, enter the name of the new geometry and select **OK**.

- 14.4.4.3 Select the certificate for the calibration standard and select the **OK** button.
- 14.4.4.4 The efficiency calibration curve will be displayed for review. Select Empirical fit, and Log scale.
- 14.4.4.5 To accept the calibration select **OK**, or select **Cancel** to abort.
- 14.4.4.6 Dismiss the Calibration report displayed to complete the calibration procedure.
- 14.4.4.7 In the DECterm type **EFFPlot**, then press ENTER the type **EFFPRINT** then hit ENTER. This will print the efficiency curve.
- 14.4.5 Efficiency Verifications
 - 14.4.5.1 Verification counts are performed as a normal sample count starting at step 15.2.3 of this SOP.
 - 14.4.5.2 No batch ID is assigned to verification counts, typically "VER" is used.
 - 14.4.5.3 Select the only sample identification available regardless of how it is named.
 - 14.4.5.3.1 You may be asked if you would like to extend the count. Select **NO**.
 - 14.4.5.3.2 When the screen to enter the sample information appears (step 15.2.8), use the date and time indicated on the manufacturer's certificate file for decay correction and change the sample identification using the following naming convention:
VER_DETECTOR_GEOMETRY, for example VER_GAM01_CAN.
 - 14.4.5.4 Once the count has completed, in the DECterm, type "@print_virtual sample, where sample equals the same identification used in step 14.4.5.3.2. This will print out the raw data of the verification count.
 - 14.4.5.5 Several pages will print out. The only pages needed are the background-subtracted peak report, which should be the first page, and the nuclide line activity report.
 - 14.4.5.6 Place the results from the "Decay Corr" column into the appropriate Master Verification Spreadsheet located at S:\RAD\FORMS\EFF_VER (where S:= sdrive on 'gelsan') under the column named **Measured Activity**.



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- 14.4.5.7 If necessary, enter the emission rate for the standard used for verification on the Master Verification Spreadsheet. This can be found on the manufacturer's certificate file for the standard. The spreadsheet will then calculate the **Calibrated Activity**. If a column for the emission rate does not exist on the spreadsheet, the **Calibrated Activity** can be calculated by using the values from the Decay Correct Source page in Alpha LIMS

(http://prodsvr01.gel.com:7778/pls/lims/de_ref_material.decay_correction).

- 14.4.5.8 The percent difference between the **Calibrated Activity** and **Measured Activity** is calculated by the spreadsheet and is displayed under the column marked **Difference**. The verification is considered acceptable if all values in the **Difference** column are less than 10%. If the **Difference** is 10% or greater, the verification is considered invalid and must be performed again. If two verifications fail notify Group Leader or Team Leader for further action.

14.5 Performance Checks

14.5.1 Daily Quality Control Calibration Check (QCC)

- 14.5.1.1 The QCC should be counted daily or prior to sample counting. If no samples are being counted this check is not required.
- 14.5.1.2 Load the QCC check source on the detector(s). If multiple QCC checks are being started skip to step 14.5.1.5.
- 14.5.1.3 From the PROcount window, select **QC | Calibration Check**.
- 14.5.1.4 Select the detector and select **OK**.
- 14.5.1.5 To start multiple QCC checks at once, select **QC | Multi Calibration Checks** from the PROcount window.
- 14.5.1.6 Highlight each detector you wish to start by clicking once on the detector name. Once you have highlighted all of the detectors you wish to start, select **OK**.

14.5.2 Daily Quality Control Background Check (QCB)

- 14.5.2.1 The QCB should be counted daily or prior to sample counting. If no samples are being counted this check is not required.
- 14.5.2.2 Ensure the detector shield(s) are empty prior to running the QCB. If multiple QCB checks are being started, skip to step 14.5.2.5.



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- 14.5.2.3 From the PROcount window, select **QC | Background Check**.
- 14.5.2.4 Select the detector and select OK.
- 14.5.2.5 To start multiple QCB checks at once, select **QC | Multi Background Checks** from the PROcount window.
- 14.5.2.6 Highlight each detector you wish to start by clicking once on the detector name. Once you have highlighted all of the detectors you wish to start, select OK.
- 14.5.3 Weekly Environmental Background
 - 14.5.3.1 Ensure the detector shield(s) is (are) empty. The same process will be used to start single and multiple weekly environmental background counts.
 - 14.5.3.2 Select **Count | Start MultipleBackgrounds** from the PROcount window.
 - 14.5.3.3 highlight each detector you wish to start by clicking once on the detector name. Once you have highlighted all of the detectors you wish to start, select **OK**.
- 14.5.4 Generating the Daily and Weekly Check Reports
 - 14.5.4.1 Daily check reports will generate every day following the completion of the QCC and QCB counts for each detector that will be in operation.
 - 14.5.4.2 In the DECterm, type the command "**@QA_REPORT D**" then hit ENTER.
 - 14.5.4.3 Weekly check reports will be completed once per week, typically Monday, following the completion of the weekly background subtraction counts.
 - 14.5.4.4 In the DECterm, type the command "**@QA_REPORT B**" then hit ENTER.

15.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

- 15.1 Prepare the sample as outlined in GL-RAD-A-013 for The Determination of Gamma Isotopes.
- 15.2 Sample Counting
 - 15.2.1 Prior to starting a sample count the detector used must be scanned into AlphaLIMS. In a web browser, enter the following address:
http://prodsvr01.gel.com:7778/pls/lims/inst_instrument.start_count
 - 15.2.2 Each sample and detector are labeled with a Universal Product Code (UPC). First scan the UPC code for the detector and then scan the UPC code for the sample. Continue doing so for any additional sample counts. Once this has been done, select Submit on the web page.



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- 15.2.3 Load the sample on the detector.
- 15.2.4 Select **Count** | **Start a Count** from the *ProCount Main Menu*.
- 15.2.5 Select a detector and select **OK**.
- 15.2.6 Enter the batch to be started and select **OK**.
- 15.2.7 Select the sample to be counted.
- 15.2.8 Enter the sample specific information into the Sample Information screen and select **OK**. (i.e., Count Time or LIMS Client Code)
- NOTE:** Sample Count Time cannot exceed the weekly BKG count time (1000 minutes)
- 15.2.9 Select the Analysis Sequence file used for analysis and select **OK**.
- 15.2.10 Select the counting geometry and select **OK**.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-010 for Counting Room Instrumentation Maintenance.

17.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

Data recording, calculation and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

18.0 POLLUTION/CONTAMINATION

Ensure all samples are bagged prior to counting to prevent instrument contamination.

19.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

Refer to GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data might require instrument maintenance, re-analysis, using a new spike mix, or a more complex set of actions. When troubleshooting measures (refer to Section 21) fail to bring an analytical process or data into control, a data exception report and/or corrective action should be initiated in accordance with GL-QS-E-004.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting the instrument is a function of analyst experience. In-house service is obtained from GEL's Group Leader or other qualified personnel. If vendor assistance is needed, then the appropriate vendor is contacted. Maintenance logbooks are kept for each instrument and contain entries for both routine and non-routine maintenance procedures.

22.0 RECORDS MANAGEMENT

- 22.1 Each sample analysis that is performed is documented in the instrument run log in accordance with GL-LB-E-009 for Run Logs.
- 22.2 All raw data printouts, calculation spreadsheets, and batch checklists are filed with the sample data for archival in accordance with GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.



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- 22.3 Instrument maintenance is recorded in accordance with GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices.
- 22.4 Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

23.0 LABORATORY WASTE HANDLING AND DISPOSAL

Laboratory waste is disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

24.0 REFERENCES

- 24.1 United States Department of Energy, Environmental Measurements Laboratory, HASL-300 The Procedures Manual of the Environmental Measurements Laboratory, 28th Edition, "Gamma Radioassay," Ga-01-R (Vol. 1), February 1997.
- 24.2 United States Environmental Protection Agency, Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Method 901.1, August 1980.
- 24.3 American National Standards Institute, American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides, ANSI N42.14-1999, with the exception of section 8, which is more applicable to the software vendor.
- 24.4 Canberra Model 480720 ProCount-ESP Users Manual, September 2000.
- 24.5 Canberra Model 480726 Genie-ESP System Users Manual, September 2000.
- 24.6 Canberra Model 480198 Genie VMS Users Manual, 2000.
- 24.7 ASTM, International, Standard Practice for Setup, Calibration, and Quality Control of Instruments Used for Radioactive Measurements, D7282-6, Nov. 2010.

25.0 HISTORY

Revision 17: Updated sections 14.4.3.6 and 14.4.5.2 for clarification.
 Revision 18: Added note to section 14.4 to clarify instrument calibration expiration dates.
 Revision 19: Revised to include new GL-RAD-D-006 for calculations.
 Revision 20: Updated Reference Section 24.3
 Revision 21: Added NOTE for sample count not to exceed weekly Bkg count time (1000 minutes)



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16-Aug-2018

SOP Effective 6/93
Revision 12 Effective March 2017

Ludlum Lucas Cell Counter

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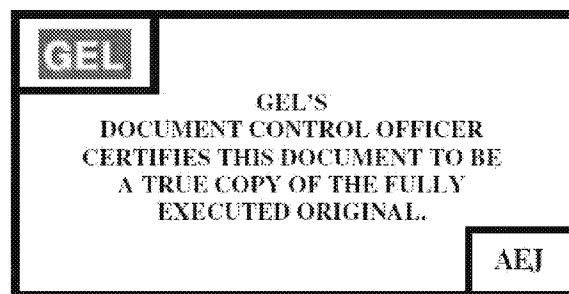
VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE
FOR
LUDLUM LUCAS CELL COUNTER

(GL-RAD-I-007 REVISION 12)

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16-Aug-2018

SOP Effective 6/93
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Ludlum Lucas Cell Counter

GL-RAD-I-007 Rev 12
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1.0 STANDARD OPERATING PROCEDURE FOR LUDLUM LUCAS CELL COUNTER

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

The purpose of this document is to give the analyst a summary of the operation of the Lucas Cell Counter.

3.0 APPLICABLE MATRIX OR MATRICES

Refer to GL-RAD-A-008 Determination of Radium-226 and GL-RAD-A-028 for Radium-226 in Drinking Water by EPA Method 903.1.

4.0 METHOD VARIATIONS

Not applicable.

5.0 DEFINITIONS

5.1 Refer to Appendix 1 for a diagram of the Ludlum front panel.

5.2 Power Switch: is a 4-position switch. "Off" indicates that the power is off. "Line" indicates that the line power is 115 volts HZ and power is on.

5.3 Count button: resets and starts the scaler counting. The scaler turns off automatically when the preset count is reached.

5.4 Count Lamp is a red light that indicates the scaler is counting.

5.5 Hold: button stops the scaler without resetting the count.

5.6 Meter Readout: displays the high voltage while the power switch is in the "line" position.

5.7 Discriminator: potentiometer is a 1-turn pot used to set the detector discriminator level. The potentiometer is set at the factory and should not be turned without first checking with the Group Leader.

5.8 High Voltage: potentiometer is used to set the detector high voltage as determined by the plateau calibration. The instrument will support up to 1500 volts.

5.9 Minutes: thumbwheel settings are 2-decade switches for setting the preset counting time. The time base is minutes from 0 to 99 with multiples of 0.1, 1 or 10.

5.10 Detector Input: is a series "C" coaxial connector used to connect the Lucas cell detector with the scaler.

5.11 Count: readout is a 6-decade LED, which indicates the total counts.

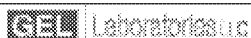
5.12 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

6.0 INTERFERENCES/LIMITATIONS

Refer to GL-RAD-A-008 for the Determination of Radium-226 and GL-RAD-A-028 for Radium-226 in Drinking Water by EPA Method 903.1.

7.0 SAFETY PRECAUTIONS AND WARNINGS

7.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan GL-LB-N-001.



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7.2 Personnel handling radioactive materials are trained in and follow the procedure outlined in the Radioactive Material Handling SOP GL-RAD-S-004.

8.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

- 8.1 Lucas Cells
- 8.2 Ludlum Model 2000 Scaler or equivalent
- 8.3 Ludlum Model 182 Radon Flask Counter

9.0 SAMPLE HANDLING AND PRESERVATION

Not applicable

10.0 REAGENTS AND STANDARDS

Not applicable

11.0 SAMPLE PREPARATION

Not applicable

12.0 QUALITY CONTROL SAMPLES

Not applicable

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE

13.1 Instrument Calibration: The instrument will be calibrated on an annual basis, or whenever the daily check indicates a condition that warrants recalibration. Calibration is performed in accordance with GL-RAD-A-008 for the Determination of Radium-226 and GL-RAD-A-028 for Radium-226 in Drinking Water by EPA Method 903.1.

13.2 Background and Efficiency Checks:

13.2.1 **Daily Instrument Efficiency Check:** Each day the instrument is used, an efficiency check is performed to ensure the consistent performance of the instrument. Place the daily source check standard in the lucas cell counter. Set the count time to 1 minute and press the "COUNT" button to begin acquisition. When the count has completed, record the total counts, analyst initials and date/time in the Lucas Cell Daily Efficiency Check Logbook. Repeat this procedure for each of the Lucas Cell Counters.

13.2.1.1 Upload the data to AlphaLIMS by selecting the "Upload Lucas Cell Daily Checks" on the Rad Macros home page. Enter the Date/Time and gross counts recorded for each detector. Press "OK" to upload the data to AlphaLIMS.

13.2.1.2 Print the daily instrument report form from AlphaLIMS. Review in accordance with GL-RAD-I-012 for Managing Statistical Data in the Radiochemistry Laboratory.

13.2.2 **Cell Background Check:** Each cell, prior to being used for analysis, is evacuated and the background counts are recorded in the cell background logbook. An administrative limit of 0.267 cpm has been set as the upper limit for the background. If the background fails, the cell may not be used for



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analysis until the cell has been purged and cleaned and demonstrated a background level of less than 0.267 cpm.

- 13.3 **Instrument Calibration:** The instrument will be calibrated in accordance with GL-RAD-A-008 or GL-RAD-A-028 on an annual basis, or whenever the daily check indicates a condition that warrants recalibration. **NOTE:** Expiration dates for annual instrument calibrations will be the last day of the month in which the calibration data is acquired.

14.0 PROCEDURE FOR ANALYSIS AND INSTRUMENT OPERATION

- 14.1 Place the Lucas Cell in the Radon Flask Counter and center the cell on the photomultiplier tube. Gently press the top portion of the flask counter down onto the base and turn the thumbscrews if available to tighten the unit in place. Allow the unit to light to adjust for a minimum of 2 minutes.
- 14.2 Select "line" operation with the power switch.
- 14.3 Select an appropriate count scale by turning the "minutes scale" as follows:
- | Time Interval | Multiplier Setting |
|-----------------|--------------------|
| 1-99 minutes | X 1 |
| 100-990 minutes | X 10 |
- 14.4 Select an appropriate count interval (typical count time is 30 minutes) by dialing in the corresponding time in minutes. (Example, for a 30 minute count dial in "3" for the left dial and "0" for the right dial with the minutes scale at X 1.)
- 14.5 Press the "count" button and release. This will reset the counter and begin the count. A red light will activate indicating that the count is in process.
- 14.6 When the count is completed, record the number present on the readout on the Queue Sheet.
- 14.7 Remove the counting cell from the counter.

15.0 EQUIPMENT AND INSTRUMENTATION MAINTENANCE

The window on the Model 182 Radon Flask Counter should be cleaned once per week.

16.0 DATA RECORDING, CALCULATION AND REDUCTION METHODS

Data recording, calculation and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

17.0 POLLUTION/CONTAMINATION

Not Applicable.

18.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

Refer to GL-RAD-D-003 for Data Review, Validation and Data Package Assembly.

19.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

- 19.1 Refer to GL-RAD-I-012 Managing Statistical Data in Radiochemistry Countroom for actions required for failed checks.
- 19.2 Refer to GL-RAD-D-003 Data Review, Validation and Data Package Assembly for actions required for failed data.



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20.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting the instrument is a function of analyst experience. In-house service is obtained from GEL Laboratories, LLC service technician. If vendor assistance is required then the appropriate vendor is contacted. Logbook entries are made to document the actions taken.

21.0 RECORDS MANAGEMENT

Daily instrument check data are recorded in the appropriate logbooks and uploaded and tracked in AlphaLIMS.

22.0 LABORATORY WASTE HANDLING AND DISPOSAL

Not Applicable.

23.0 REFERENCES

23.1 Ludlum Model 182 Radon Flask Counter Operating Manual.

23.2 Ludlum Model 2000 Scaler Operations Manual.

24.0 HISTORY

Revision 9: Updated sections 13.0 and 14.0 to give a more adequate description of the process.

Revision 10: Note added in section 13.3 to clarify instrument calibration expiration dates.

Revision 11: Updated procedure reference to GL-RAD-D-006 for Equations Used in Data Reduction for Environmental Radiochemistry.

Revision 12: Updated to add calibration and typical count time.

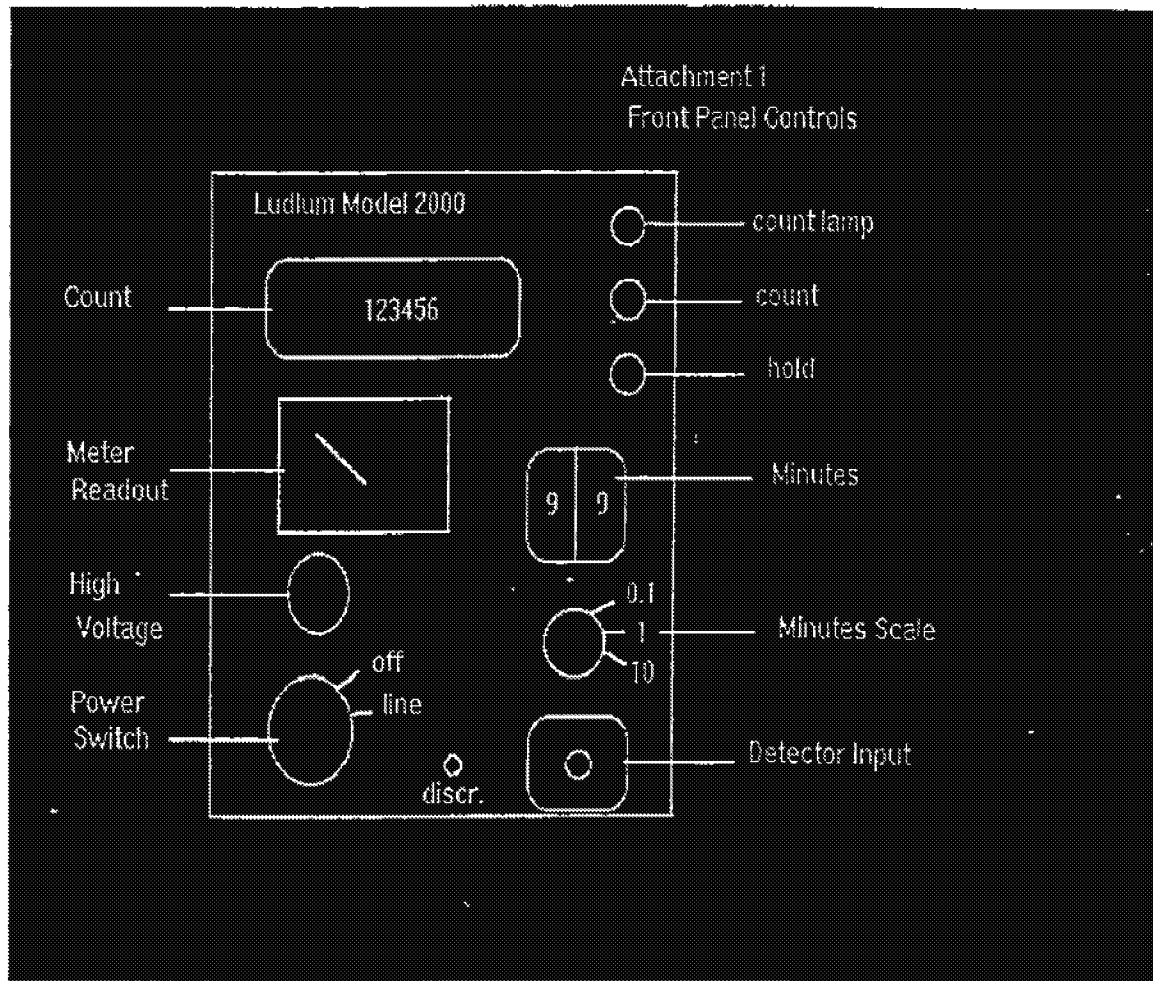


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APPENDIX 1

LUDLUM MODEL FRONT PANEL



16-Aug-2018

Standard Operating Procedure for Alpha Spectroscopy System
SOP Effective Date 6/10/93
Revision 15 Effective May 2015

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VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE
FOR
ALPHA SPECTROSCOPY SYSTEM

(GL-RAD-I-009 REVISION 15)

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1.0 STANDARD OPERATING PROCEDURE FOR ALPHA SPECTROSCOPY SYSTEM**2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY**

This method establishes the procedures for general use and calibration of the Canberra Alpha Spectroscopy System used to obtain and analyze alpha spectra for samples containing single or multiple alpha-emitting radionuclides. The operation of the Canberra Alpha Analyst and model 7401 alpha spectrometers is discussed. This method also describes how specific radionuclides are identified and quantified from the spectral data.

This procedure also outlines the required scheduled maintenance and performance checks for the instruments. In order to assure the optimum performance of count room instrumentation, it is necessary to perform regularly scheduled maintenance and instrument checks. The instrument checks include energy and efficiency calibration, which are conducted once a month, backgrounds, which are conducted weekly, and daily pulser checks. The scheduled maintenance provides a means of maintaining instrument performance, while minimizing the "down time" due to instrument failure and repair.

3.0 APPLICABLE MATRICES

Applies to all matrices.

4.0 METHOD SCOPE, APPLICABILITY AND DETECTION LIMIT

The procedure is not specific to one particular method. For method scope, applicability or detection limit refer to the method specific analytical standard operating procedure.

5.0 METHOD VARIATIONS

Not applicable.

6.0 DEFINITIONS

- 6.1 Average Efficiency: The average of the calculated efficiency of each isotope contained on the efficiency standard.
- 6.2 Background: Those counts that can be observed and thereby, allowed for by measuring a blank background planchet. These counts are attributable to environmental radioactivity, recoil contamination of the detector, electronic noise pulses, etc.
- 6.3 Efficiency: A percent of decay events from a standard radioactive source that are seen and measured by a detector.
- 6.4 Energy Calibration Offset: The energy (keV) that corresponds to the first channel on the Multichannel Analyzer for each chamber.
- 6.5 FWHM (Full Width Half Maximum): The full width of an alpha peak distribution measured at half the maximum peak height.
- 6.6 Peak Area: The number of counts contained within an alpha peak.
- 6.7 Peak Energy: The energy (keV) measured at the center of the alpha peak.

- 6.8 Peak Resolution: The FWHM value of the alpha peak.
- 6.9 Performance Check: Any operation performed on an instrument to verify its ability to conform to required specifications
- 6.10 PIPS detector: Passivated Implanted Planar Silicon
- 6.11 Scheduled Maintenance: Any operation performed on an instrument to prevent premature equipment failure
- 6.12 Traceable Calibration Standard: A calibrated radioactive source, with stated accuracy, whose calibration is certified by or to NIST (National Institute of Standards and Technology) or an equivalent organization.
- 6.13 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.
- 6.14 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.

7.0 INTERFERENCES/LIMITATIONS

For analyses requiring isotope specific analyses (i.e. U-238, Pu-238) chemical separations are performed during sample preparation to remove unwanted counting interferences.

8.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

- 8.1 Refer to the Radioactive Material Handling Procedure (GL-RAD-S-004) for instructions on the handling of radioactive samples.
- 8.2 Refer to the Laboratory Waste Management Plan (GL-LB-G-001) for instructions on proper disposal of materials.
- 8.3 The detector bias supply must remain off, until the detector chamber reaches the normal operating vacuum, to prevent damage to the surface barrier detectors.
- 8.4 Turning off, or loss of power to, the vacuum pumps could lead to oil contamination of the alpha detectors. Therefore, all detectors must be brought to atmospheric pressure prior to turning the vacuum system off or immediately after a loss of power.
- 8.5 Follow the manufacturer's instructions for set up, intercomponent connections, and preliminary testing of the equipment. Observe all of the manufacturer's limitations and precautions.
- 8.6 Never exceed the manufacturer's recommended operating voltage for the detector; this may lead to detector damage.

9.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

- 9.1 Canberra model 7401 Alpha Spectrometer
- 9.2 Canberra model 7200 Dual Alpha Analyst Spectrometer

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| <p>Standard Operating Procedure for Alpha Spectroscopy System</p> <p>SOP Effective Date 6/10/93</p> <p>Revision 15 Effective May 2015</p> | <p>GL-RAD-I-009 Rev 15</p> <p>Page 5 of 19</p> |
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- 9.3 Canberra model 7200 Controller
- 9.4 DEC/Compaq Alpha workstation or equivalent
- 9.5 Vacuum Pump
- 9.6 AMX analog multiplex or module
- 9.7 Acquisition Interface Module
- 9.8 ADC (analog to digital converter)
- 9.9 Vacuum pump and filtration rig
- 9.10 Disposable filter funnels (containing 25 µm filters with 0.1 mm pore size)
- 9.11 Stainless steel disks (29 mm diameter)
- 9.12 Stainless steel tweezers

10.0 REAGENTS AND STANDARDS

- 10.1 Traceable Calibration Standard - The actual standard is dependent upon the sample geometry being calibrated.
- 10.2 Vacuum Pump Oil
- 10.3 Silicone grease

11.0 SAMPLE HANDLING AND PRESERVATION

Not applicable.

12.0 SAMPLE PREPARATION

Not applicable.

13.0 QUALITY CONTROL SAMPLES

Not applicable.

14.0 STANDARDIZATION AND CALIBRATION

NOTE: Refer to GL-RAD-M-001 for guidance on the preparation of calibration sources for alpha spectroscopy.

- 14.1 Energy and Efficiency Calibration (Monthly checks for Alpha analyst detectors)
 - 14.1.1 Alpha calibration standards are counted once each calendar month to update the detector energy and efficiency calibrations.
 - 14.1.2 From the AMS Procedure window, select Displays then Chamber Status; ensure detectors are free for use.
 - 14.1.3 Using a pair of tweezers, carefully position the appropriate calibration standard into each counter, taking care to center the calibration standard beneath the detector face and ensure the sample shelf is in the proper location.

- 14.1.4 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.
- 14.1.5 Close the chamber doors.
- 14.1.6 From the AMS Procedure window, select **Count** then **Primes**.
- 14.1.7 Enter a list of the chambers that you are starting and click **OK** or press **Enter**.
- 14.1.8 The detectors will automatically evacuate air from the chamber and apply a detector bias. The system then starts data acquisition on all alpha counters and counts the standards for a pre-determined time suitable to achieve greater than 10,000 counts in each applicable region of interest (Gd-148, Np-237, and Cm-244) typically 2 to 4 hours.
- 14.1.9 When the count is complete, the detector bias will automatically be turned off and the chamber vented to atmosphere.
- 14.1.10 When the calibration count is completed, proceed with section 14.3.
- 14.2 Energy and Efficiency Calibration (Monthly checks for model 7401 detectors)
 - 14.2.1 Alpha calibration standards are counted once each calendar month to update the detector energy and efficiency calibrations.
 - 14.2.2 From the DECTerm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting System Main Menu is displayed, proceed with 14.2.3. If the Sample Counting System Main Menu is not displayed, consult with the Group Leader or their designee.
 - 14.2.3 Select **1) Sample Counting** to access the Sample Counting Menu.
 - 14.2.4 From Sample Counting Menu, Select **2) List Status of Detectors**; ensure detectors are free for use. Press **Return** to exit.
 - 14.2.5 Using a pair of tweezers, carefully position the appropriate calibration standard into each counter, taking care to center the calibration standard beneath the detector face and ensure the sample shelf is in the proper location.
 - 14.2.6 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.
 - 14.2.7 Close the chamber doors and start evacuation of the chambers in accordance with section 15.5.
 - 14.2.8 After normal operation vacuum is achieved, turn on detector bias.
 - 14.2.9 Select **1) Count a New Sample** to access the Alpha Counting Menu.

- 14.2.10 Select **3) Monthly Calibration Check**.
- 14.2.11 Verify that all detector bias supplies are on, all pulsers are off. Enter a list of detectors to start, and press **Return**. The system then starts data acquisition on all alpha counters and counts the standards for a pre-determined time suitable to achieve greater than 10,000 counts in each applicable region of interest (Gd-148, Np-237, and Cm-244) typically 2 to 4 hours.
- 14.2.12 When the calibration count is completed, proceed with section 14.3.
- 14.3 Manually Processing the Monthly Calibrations
 - 14.3.1 Proceed to DECterm VMS prompt. If the sample Counting System menu is still displayed on the DECterm, then exit to the DECterm VMS prompt by pressing **R** and **Return**.
 - 14.3.2 Check the contents of the file **NAMES.DAT** by typing **EDIT NAMES.DAT**. Edit the contents of the file so it contains the entries **W###-W###**, depending on the detectors to be processed. When the contents of the file are correct, press **Control Z** followed by **Quit** if the changes are not to be saved, or **Exit** if the changes are to be save. Type **RANGE # #** to indicate which banks to show on the supervisor's action report.
 - 14.3.3 At the prompt, type **Process** to start the processing of the calibrations.
 - 14.3.4 At the **Initial or Update calibration? (I/U)** prompt, enter **U** to update the calibration or enter **I** to perform an initial calibration. Note that an initial calibration is done only under specific circumstances, such as initial setup of a counter. Consult the Group Leader or designee prior to performing an initial calibration. If a calibration update was chosen, the operator will be asked to verify the update energy and efficiency parameters for each detector.
 - 14.3.5 Initial Calibration Only
 - 14.3.5.1 At the prompt, use the mouse to position the cursor over the center of the specified nuclide on the spectrum display. Press **Return**.
 - 14.3.5.2 Repeat for each nuclide.
 - 14.3.6 Proceed with section 14.4.
- 14.4 Reviewing Monthly Calibration Report
 - 14.4.1 After the monthly calibration data is processed, a supervisor's action report will be printed. Review the report for any out of control condition. Contact Group Leader or designee for out of control conditions. Detector should be removed from service if any of the following conditions exist: ABOVE/BELOW PSAREA,

NLACTIVITY, ECOFFSET, ECSLOPE, AVRGEFF, FWHMCONST, PSENERGY, PSFWHM.

NOTE: Refer to GL-RAD-I-012, Managing Statistical Data in the Radiochemistry Laboratory, for guidance on locking out detectors.

14.4.2 Update detector status board.

14.5 Daily Pulser Checks for Alpha analyst detectors

14.5.1 Daily pulser checks performed daily, prior to counting samples, to verify the proper operation of the detectors. Peak centroid, Pulser count rate, and peak FWHM are monitored and stored in quality assurance files.

14.5.2 From the AMS Procedure window, select **Displays** then **Chamber Status**; ensure detectors are free for use.

14.5.3 From the AMS Procedure window, select **Count** then **Pulsers**.

14.5.4 Enter a list of the chambers that you are starting and click **OK** or press **Enter**.

14.5.5 The detectors will automatically evacuate air from the chamber and apply a detector bias. The system then starts data acquisition on all alpha counters and counts the pulsers for 5 minutes.

14.5.6 When the count is complete, the detector bias will automatically be turned off and the chamber vented to atmosphere.

14.5.7 When the daily pulser count is completed, the data may be automatically processed and a Supervisor's Action Report is generated. If a report is generated, proceed with section 14.8. If a report is not generated, proceed with section 14.7.

14.6 Daily Pulser Checks for model 7401 detectors

14.6.1 Daily pulser checks performed daily, prior to counting samples, to verify the proper operation of the detectors. Peak centroid, Pulser count rate, and peak FWHM are monitored and stored in quality assurance files.

14.6.2 From the DECterm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting System Main Menu is displayed, proceed with 14.6.3. If the Sample Counting System Main Menu is not displayed, consult the Group Leader or their designee.

14.6.3 Select **1) Sample Counting** to access the Sample Counting Menu.

14.6.4 From Sample Counting Menu, **Select 2) List Status of Detectors**; ensure detectors are free for use. Press **Return** to exit.

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- 14.6.5 If needed start evacuation of the chambers in accordance with section 15.5.
- 14.6.6 After normal operating vacuum is achieved, turn on detector bias and activate the detector pulser.
- 14.6.7 Select **1) Count a New Sample** to access the Alpha Counting Menu.
- 14.6.8 Select **2) Daily Pulser Check**.
- 14.6.9 Verify that all pulser and bias supplies are enabled for each detector. Enter a list of detectors to start. The system then starts data acquisition on all alpha counters and counts the pulsers for 5 minutes.
- 14.6.10 When the daily pulser count is completed, the data may be automatically processed and a Supervisor's Action Report generated. If a report is generated, proceed with section 14.8. If a report is not generated, proceed with section 14.7.
- 14.7 Manually Processing the Daily Pulser Checks
 - 14.7.1 Proceed to the DECterm VMS prompt. If the Sample Counting System menu is still displayed on the DECterm, then exit to the DECterm VMS prompt by pressing **R** and **Return**.
 - 14.7.2 Check the contents of the file **NAMES.DAT** by typing **EDIT NAMES.DAT**. Edit the contents of the file so it contains the entries **D###-D###**, depending on the number of detectors to be processed. When the contents of the file are correct, press **Control Z** followed by **Quit** if the changes are not to be saved, or **Exit** if the changes are to be saved. Type **RANGE ##** to indicate which banks to show on the supervisor's action report.
 - 14.7.3 At the \$ prompt, type **Process** to start the processing of the Daily Pulser Checks. The program proceeds to automatically process the pulser data one counter at a time.
 - 14.7.4 Proceed with section 14.8.
- 14.8 Reviewing Daily Pulser Report
 - 14.8.1 After the data is processed, a Supervisor's Action Report and Missing QA Report will be printed. Review the Supervisor's Action Report for any out of control conditions. Contact Group Leader or designee immediately for out of control conditions. Detector should be removed from service for the day if any of the following conditions exist: ABOVE/BELOW PSFWHM, PSENERGY, PSCENTRD, PSCTSS. Review the Missing QA Report for any detectors that may not have run Daily Pulser Checks. Perform section 14.6 or 14.5 as needed on any detectors listed on the Missing QA Report.

NOTE: Refer to GL-RAD-I-012, Managing Statistical Data in the Radiochemistry Laboratory, for guidance on locking out detectors.

14.8.2 Update detector status board.

14.9 Weekly Background Checks for Alpha Analyst detectors

14.9.1 Blank planchets are counted once each week to update the detector background counts.

14.9.2 From the AMS Procedure window, select **Displays** then **Chamber Status**; ensure detectors are free for use.

14.9.3 Using a pair of tweezers, carefully position the appropriate background planchets into each counter, taking care to center the planchet beneath the detector face and ensure the sample shelf is in the proper location.

14.9.4 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.

14.9.5 Close the chamber doors.

14.9.6 From the AMS Procedure window, select **Count** then **Backgrounds**.

14.9.7 Enter a list of the chambers that you are starting and click **OK** or press **Enter**.

14.9.8 The detectors will automatically evacuate air from the chamber and apply a detector bias. The system then starts data acquisition on all alpha counters and counts the backgrounds for the predetermined count time.

14.9.9 When the count is complete, the detector bias will automatically be turned off and the chamber vented to atmosphere.

14.9.10 When the background is completed, the data may be automatically processed and a Supervisor's Action Report generated. If a report is generated, proceed with section 14.12. If a report is not generated, proceed with section 14.11.

14.10 Weekly Background Checks for model 7401 detectors

14.10.1 Blank planchets are counted once each week to update the detector background counts. Weekly backgrounds are counted for 1000 minutes.

14.10.2 From the DECterm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting System Main Menu is displayed, proceed with 14.10.3. If the Sample Counting System Main Menu is not displayed, consult the Group Leader or their designee.

14.10.3 Select **1) Sample Counting** to access the Sample Counting Menu.

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- 14.10.4 From Sample Counting Menu, Select **2) List Status of Detectors**; ensure detectors are free for use. Press Return to exit.
- 14.10.5 Using a pair of tweezers, carefully position the appropriate background planchets into each counter, taking care to center the planchet beneath the detector face and ensure the sample shelf is in the proper location.
- 14.10.6 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.
- 14.10.7 Close the chamber doors and start evacuation of the chambers in accordance with section 15.5.
- 14.10.8 After normal operating vacuum is achieved, turn on detector bias.
- 14.10.9 Select **1) Count a New Sample** to access the Alpha Counting Menu.
- 14.10.10 Select **3) Backgrounds**.
- 14.10.11 Verify that all detector bias supplies are on, all pulsers are off. Enter a list of detectors to start, and press **Return**. The system then starts data acquisition on all alpha counters and counts the backgrounds for the predetermined count time.
- 14.10.12 When the background count is completed, the data may be automatically processed and a Supervisor's Action Report generated. If a report is generated, proceed with section 14.12. If a report is not generated, proceed with section 14.11.
- 14.11 Manually Processing Weekly Backgrounds
 - 14.11.1 Proceed to the DECterm VMS prompt. If the Sample Counting System menu is still displayed on the DECterm, then exit to the DECterm VMS prompt by pressing **R** and **Return**.
 - 14.11.2 Check the contents of the file **NAMES.DAT** by typing **EDIT NAMES.DAT**. Edit the contents of the file so it contains the entries **B###-B###**, depending on the number of detectors to be processed. When the contents of the file are correct, press **Control Z** followed by Quit if the changes are not to be saved, or Exit if the changes are to be saved. Type **RANGE # #** to indicate which banks to show on the supervisor's action report.
 - 14.11.3 At the \$ prompt, type **Process** to start the processing of the background counts. The program proceeds to automatically process the background data one detector at a time.
- 14.12 Reviewing Weekly Background Report
 - 14.12.1 After the data is processed, a Supervisor's Action Report will be printed. Review the printout for any out of control conditions. Contact

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Group Leader or designee immediately for out of control conditions.
Detector should be logged out for isotopes that have a high background.

NOTE: Refer to GL-RAD-I-012, Managing Statistical Data in the Radiochemistry Laboratory, for guidance on locking out detectors.

14.12.2 Update detector status board.

15.0 OPERATING PROCEDURE

15.1 Sample Counting (Alpha Analyst detectors)

- 15.1.1 From the AMS Procedure window, select **Displays** then **Chamber Status**; ensure detectors are free for use.
- 15.1.2 Open the door of the sample chamber. Carefully remove any sample that is in the chamber with a pair of tweezers and place it in a storage container.
- 15.1.3 Using a pair of tweezers, carefully position the next sample that is to be counted on the sample shelf, taking care to center the sample beneath the detector face and ensure the sample shelf is in the proper location.
- 15.1.4 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.
- 15.1.5 Close the chamber doors.
- 15.1.6 From the AMS Procedure window, select **Count** then **Samples**.
- 15.1.7 Enter a list of the chambers that you are starting and click **OK** or press **Enter**.
- 15.1.8 A window will appear indicating all detectors have started. Click **OK**.
- 15.1.9 The detectors will automatically evacuate air from the chamber and apply a detector bias. The system starts data acquisition on all alpha counters. The default count time is set to four (4) hours, but can be changed during step 15.3.4 if necessary.
- 15.1.10 If data acquisition is to be started on model 7401 detectors at the same time, proceed to section 15.2.
- 15.1.11 If only Alpha Analyst detectors are to be started, return to the DECterm window before proceeding further.
- 15.1.12 From the DECterm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting System Main Menu is displayed, proceed with 15.1.13. If the Sample Counting System Main Menu is not displayed, consult the Group Leader or their designee.
- 15.1.13 Select **1) Sample Counting** to access the Sample Counting Menu.

- 15.1.14 Select **1) Count a New Sample** to access the Alpha Counting Menu.
- 15.1.15 Select **1) Samples**.
- 15.1.16 At the “Use which detector bank to count (RETURN to end)”: prompt, press **Return**.
- 15.1.17 Enter sample information for each sample in accordance with section 15.3.
- 15.2 Sample Counting (model 7401 detectors)
 - 15.2.1 From the DECterm VMS prompt, type **Count** to access the Sample Counting System Main Menu. If the Sample Counting Main Menu is displayed, proceed with 15.2.2. If the Sample Counting System Main Menu is not displayed, consult the Group Leader or their designee.
 - 15.2.2 Select **1) Sample Counting** to access the Sample Counting Menu.
 - 15.2.3 From Sample Counting Menu, Select **2) List Status of Detectors**; ensure detectors are free for use. Press **Return** to exit.
 - 15.2.4 Ensure that there are no jobs active which use the alpha spectrometer(s) that is to be loaded. Ensure the bias supply to the detector is off, and then vent the chambers in accordance with section 15.5.
 - 15.2.5 Open the door of the sample chamber. Carefully remove any sample that is in the chamber with a pair of tweezers and place it in a proper storage container.
 - 15.2.6 Using a pair of tweezers, carefully position the next sample that is to be counted on the sample shelf, taking care to center the sample beneath the detector face and ensure the sample shelf is in the proper location.
 - 15.2.7 Inspect the vacuum seal on the chamber door to ensure that no debris exists that may interfere with vacuum pressure. Clean the seal with a dry lint free cloth if necessary.
 - 15.2.8 Close the chamber doors and start evacuation of the chambers in accordance with section 15.5.
 - 15.2.9 After normal operating vacuum is achieved, turn on detector bias.
 - 15.2.10 Before starting acquisition, verify the proper operating bias.
 - 15.2.11 Select **1) Start a New Count** to access the Alpha Counting Menu.
 - 15.2.12 Select **1) Samples**.
 - 15.2.13 Enter the bank of detectors to acquisition on (or press **Return** when all needed banks are on) and the sample count time. Repeat for each bank as needed.
 - 15.2.14 Enter sample information for each sample in accordance with section 15.3.

15.3 Entering Sample Information

- 15.3.1 Enter the Batch ID (or press **Return** when all count data has been entered and proceed to 15.3.6). The system will gather batch information from AlphaLIMS if this is the first time the batch number has been entered. If the system appears to hang while “Getting batch information from AlphaLIMS” is displayed, type **Control-Y** to skip to the next step. (In this case, no default information will be provided.)
- 15.3.2 Enter/verify appropriate batch parameters. Press **PF1** to accept the batch parameters and exit the Parameter Editor.
- 15.3.3 Enter the detector number containing the sample. If all samples for the batch have been entered, type **P** (if the batch is ready to proceed to data review) or **C** (if batch is remaining in the count room) and press **RETURN**. If there are more batches to count, continue with section 15.3.1. If no other batches are to be started press **RETURN** and go to section 15.3.6.
- 15.3.4 The system then enters the Parameter Editor. Enter/verify the displayed information for the sample.
- 15.3.5 Press **PF1** to exit the Parameter Editor. Continue with 15.3.3 for the next sample.
- 15.3.6 Monitor detectors for high count rates. If the count rate exceeds 100 counts per minute (across the entire spectrum), turn off the bias, vent the chamber, remove the sample from the chamber, and contact the Group Leader (or designee) immediately. A one-hour background count may be necessary to confirm that the detector was not contaminated.
- 15.3.7 When sample counts are finished, each sample may be processed automatically. If spectrum data needs to be manually processed, proceed with section 15.4.

15.4 Processing Sample Data via AlphaGEL

- 15.4.1 This section assumes the operator has a general understanding of the Microsoft Windows operating environment.
- 15.4.1.1 AlphaGEL is a custom software package developed exclusively for GEL Laboratories, which runs from a PC.
- 15.4.1.1.1 Start the client program from the Windows start menu or the desktop icon.
- 15.4.1.1.2 If necessary, click “Network”, then “Connect” to connect to the AlphaGEL server. Optionally, double-click on the red network

status indicator. Enter Username and Password if necessary.

- 15.4.2 Enter the batch number of the samples to process in the main box of the "Processing" frame of the "AlphaGEL Remote Connection-Client" window and click "View Batch Info" or press **Enter**.
- 15.4.3 Verify all sample information in the "Batch Information" window.
 - 15.4.3.1 Sample information can be corrected by either double-clicking a cell or selecting a cell and pressing Enter.
 - 15.4.3.2 Double-clicking the column header can change an entire column. This will cause all values in the column to be the same.
 - 15.4.3.3 Sample information changes are not saved until "Confirm All Changes" is clicked.
 - 15.4.3.4 If changes are to be discarded, clicking "Tools", "Revert to Saved" will restore the information to the last saved state.
 - 15.4.3.5 Verify that the radioactive standards are correct by clicking "Standard" and reviewing the "Standards Information" window.
- 15.4.4 Mark the samples from the batch to process by using the left-hand column. "X" indicates that the sample will be processed. All samples are marked for processing when the batch is first opened.
- 15.4.5 Verify that the "Client Processing Options" are correct. If any changes to these needs to be made, consult with the Group Leader or their designee.
- 15.4.6 Click "Process this Batch" to begin processing the marked samples.
- 15.5 Vacuum pump operation for model 7401 detectors
 - 15.5.1 Evacuation of Air from Alpha Spectrometer model 7401
 - 15.5.1.1 Ensure that the vacuum manifold control is set to the "**pump down manifold**" position.
 - 15.5.1.2 Place the "**pump/vent**" valve/switch on the spectrometer in the "**pump and locked**" position.
 - 15.5.1.3 Repeat 15.5.1.2 for each chamber in the bank.
 - 15.5.1.4 Monitor the pump down manifold vacuum gauge until the indicator is below 10 millimeters of mercury.
 - 15.5.1.5 Place the vacuum manifold control in the "**high vacuum manifold**" position. The detectors are now at normal operating vacuum.

15.5.2 Venting Alpha Spectrometer model 7401 to Atmosphere

15.5.2.1 Ensure that the vacuum manifold control is set to the **"pump down manifold"** position.

15.5.2.2 Place the **"pump/vent"** valve/switch on the spectrometer in the **"vent"** position.

15.5.2.3 Repeat 15.5.2.2 for each chamber in the bank.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-010 Counting Room Instrument Maintenance.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Refer to GL-RAD-D-003 Data Review, Validation, and Data Package Assembly.

18.0 POLLUTION/CONTAMINATION

Not applicable.

19.0 DATA RECORDING, CALCULATIONS, AND REDUCTION METHODS

Data recording, calculation, and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

20.0 CORRECTIVE ACTION FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data might require instrument maintenance, re-analysis, using a new spike mix, or a more complex set of actions. When troubleshooting measures fail to bring an analytical process or data into control, a Data Exception Report (DER) and/or corrective action should be initiated in accordance with AlphaLIMS Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, GL-QS-E-004 and/or Conducting Corrective/Preventive Action and Identifying Opportunities for Improvement, GL-QS-E-002.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting the instrument is a function of analyst experience. In-house service is obtained from GEL's Group Leader or other qualified personnel. If vendor assistance is needed, then the appropriate vendor is contacted. Maintenance logbooks are kept for each instrument and contain entries for both routine and non-routine maintenance procedures.

22.0 RECORDS MANAGEMENT

22.1 Each sample analysis that is performed is documented in the instrument run log in accordance with GL-LB-E-009 Run Logs.

22.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival in accordance with GL-RAD-D-003 Data Review, Validation, and Data Package Assembly.

22.3 Instrument maintenance is recorded in accordance with GL-LB-E-008, Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices.

23.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Refer to GL-LB-G-001 Laboratory Waste Management Plan.

24.0 REFERENCES

- 24.1 1990 Annual Book of ASTM Standards, Volume 12.02, E181.
- 24.2 Canberra Model 7401 Alpha Spectrometer Operations Manual.
- 24.3 U.S. Department of Energy Quality Systems for Analytical Services (DOE QSAS), Revision 2.8, January 2012
- 24.4 Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP), Chapter 18, July 2004
- 24.5 American Society for Testing and Materials (ASTM), ASTM C 1128-01(2008) and C1159-03 (2003).
- 24.6 National Institute of Science and Technology (NIST), Technical Note 1297, 1994.

25.0 HISTORY

Revision 15: Updated to correct grammatical errors as well as for formatting issues.
Revision 14: Updated to comply with current process as part of annual review.
Revision 13: Added reference note for the preparation of calibration sources for alphaspectrometer. Removed guidelines for the preparation and recertification of rare-earth fluoride efficiency sources for alpha spectrometry.
Revision 12: Added section for processing samples via AlphaGEL.
Revision 11: Annual review: Updated SOP throughout.

APPENDIX 1: TROUBLESHOOTING

| Symptom | Possible Cause | Solution |
|--|---------------------------------|--|
| Vacuum light on Alpha Analyst pair continuously blinks (beyond normal pump down time) | Vacuum pump oil low | Check the oil level on the vacuum pump and fill if necessary. |
| | Debris on vacuum seal of door | From the AMS Procedure window, select Count then Pause Acquire . Select AA then Vent Chambers . Open both chambers & clean the seal with a dry lint free cloth. Close the chambers. From the AMS Procedure window, select AA then Pump Chambers . Select Count then Unpause Acquire . |
| Acquisition of Alpha Analyst detector paused | Chamber was manually paused | From the AMS Procedure window, select Count then Unpause Acquire . Enter the detector to unpause. |
| | Vacuum leak to chamber | See "Vacuum light on Alpha Analyst pair continuously blinks" for possible solutions. |
| | Electronic fault | Check to see if the "fault" light on the detector is on, if so, abort acquisition by selecting Count then Abort Acquire from the AMS Procedure window for the effected chambers. Restart the count in accordance with section 14.1, 14.5, 14.9, or 15.1 as appropriate. |
| Buttons and/or switches on model 7401 detector do not appear to be functioning | Multiple causes | Reset chamber by simultaneously pressing RESET and DIGIT SELECT and move the INC/DEC switch to INC. Release the buttons & switch when the display goes blank. |
| Apparent poor vacuum (7401 only) indicated by display (above 800 μ m Hg) or vacuum gauge | Vacuum sensor of chamber is bad | If the majority of other detectors on the same vacuum pump indicate a proper vacuum, the vacuum sensor in the detector is likely malfunctioning. This will not hinder detector operation. |
| | Vacuum pump oil low | Check the oil level on the vacuum pump and fill if necessary. |
| | Debris on vacuum seal of door | Attempt to isolate the bank where the poor seal is by putting manifold controls to hold position for each bank on the same pump to see if the vacuum shows improvement. Once a specific bank is isolated, use the chamber vacuum controls to further isolate the individual chamber. Once a single chamber is isolated as the cause, open the chamber & clean the seal with a dry lint free cloth. Restart the count of the affected bank. |

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| | Manifold valve leaking | Attempt to isolate the bank where the leak is by putting manifold controls to hold position for each bank on the same pump to see if the vacuum shows improvement. Once a specific bank is isolated, check that the seal of the manifold valve is tight. Remove the valve lever by loosening the screw under the larger end & tighten the nut around the manifold valve. Be sure the nut is not so tight that the valve cannot be turned. Replace the valve lever. |
| | Leaking or cracked vacuum hose | Inspect hoses for loose connections or cracks. Replace cracked hose if necessary. If seal is loose, tighten hose clamp and/or add vacuum grease. |
| | Cracked or worn vacuum connection inside chamber | This problem involves working close to the electronics inside a 7401 detector. Consult the Group Leader (or designee) regarding repair of this condition. |
| Vacuum control knob loose (7401 only) | Alan screw loose | Using an alan wrench, tighten the two screws inside the knob. |
| DECterm window is not displayed | Window closed or computer was restarted | Open a new DECterm window from the Session Manager toolbar by selecting Applications then DECterm . |
| AMS Procedure window is not displayed | Window closed or computer was restarted | Open a new AMS workspace from the Spectroscopy Assistant window by selecting File then Open Workspace . Select the file HUME.WSP then click OK . |
| Spectroscopy Assistant window is not displayed | Window closed or computer was restarted | Open a new Spectroscopy Assistant window from the Session Manager toolbar by selecting Applications then AMS Spec. Assistant . |

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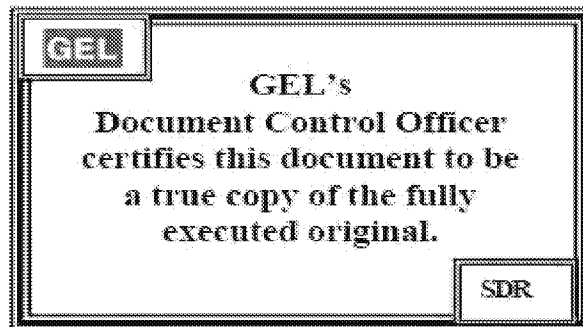
VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE
FOR
COUNTING ROOM INSTRUMENTATION
MAINTENANCE

(GL-RAD-I-010 REVISION 20)

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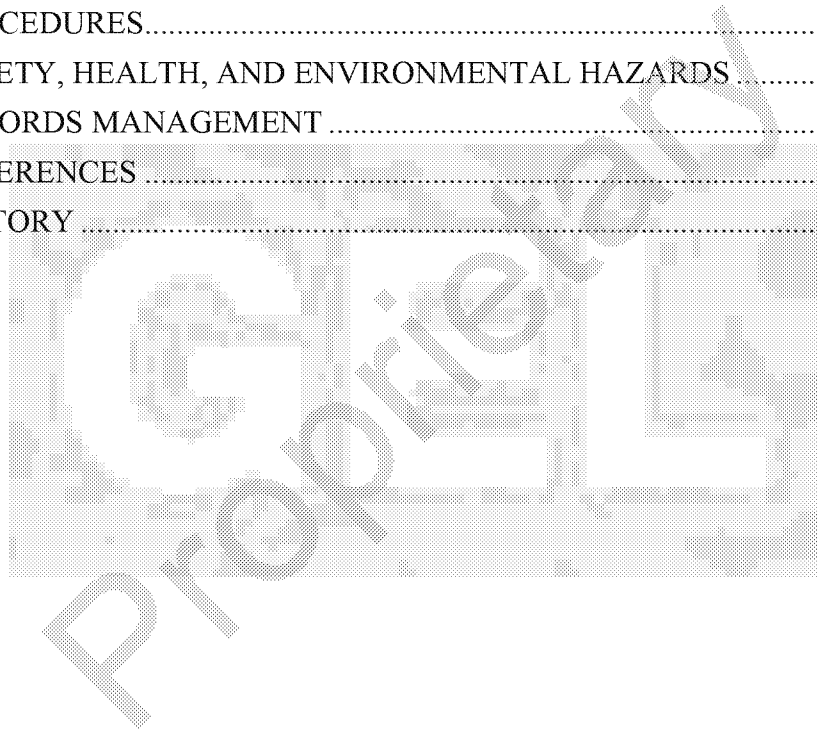
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1.0 STANDARD OPERATING PROCEDURE FOR COUNTING ROOM INSTRUMENTATION MAINTENANCE

2.0 PURPOSE

This procedure outlines the routine maintenance for each of the counting room instruments.

3.0 DISCUSSION

In order to assure the optimum performance of counting room instrumentation, it is necessary to perform routine maintenance. The routine maintenance provides a means of maintaining instrument performance while minimizing the "down time" due to instrument failure and subsequent repair.

4.0 DEFINITIONS

- 4.1 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.
- 4.2 Routine Maintenance: Any operation performed on an instrument to prevent premature equipment failure or to eliminate or minimize instrument contamination.

5.0 PROCEDURES

5.1 Gamma Spectrometers

5.1.1 Liquid nitrogen fill – Each detector must be kept cold to ensure proper operation. Keeping the Dewars filled with liquid nitrogen does this.

5.1.1.1 Safety precautions – Due to the nature of liquid nitrogen (approximately -320° F) safety goggles and cryogenic gloves are recommended. Also, when the Dewars vent, nitrogen gas can displace oxygen in the air. Before filling, open the main door between the count room and the Prep Laboratory to increase the flow of air into the count room. Oxygen monitors are present in the count room. If any of the alarms on the monitors are triggered, leave the area and contact the Group Leader or other designated personnel for further instructions.

5.1.1.2 Dewar fill – Three stages of valves are used in the Dewar fill procedure: the main count room valve, the line valves, and the individual detector valves. Once the fill has begun, one individual detector valve on a line must always be open to ensure that pressure does not build up in the pipes. To begin, open the line valve and the individual valves for any detectors to be filled. Close the line valves for the detectors that are not going to be filled. Open the main valve to begin allowing liquid nitrogen to fill the lines.

5.1.1.3 Finishing the Dewar fill – As the Dewars fill (indicated by liquid nitrogen exiting from the exhaust hoses on the

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Dewars) close off their valves until only one valve remains on a line. Once the last Dewar on a line is filled, do not close off that individual valve. Instead, close off the line valve. If only one line valve remains open with only one Dewar left to fill, do not close off the line valve or the individual valve. Instead, close off the main count room valve. This will ensure that a closed pressure system is not created.

5.1.1.4 Wait a minimum of one hour to resume counting to allow the detectors to adjust and to minimize any thermal excitation that may occur from the Dewar fill.

5.1.2 Software Backups – Refer to section 5.6 for VMS backup system.

5.2 Alpha Spectrometry System

5.2.1 Software Backups – Refer to section 5.6 for VMS backup system.

5.2.2 Vacuum Pump Oil – The oil in the alpha spectrometry system vacuum pumps shall be changed as a minimum of semi-annually.

5.2.3 Filter Cleaning – The filter on the air intake of the instrument cabinet shall be cleaned as a minimum quarterly.

5.3 Gas Flow Proportional Counters

5.3.1 Software Backups – Data are archived on a remote server that is backed up/maintained by the CST Department.

5.3.2 Sample Shelf Cleaning – The sample shelf assembly should be cleaned periodically to minimize the accumulation of contamination. This is best performed prior to running the weekly and/or daily backgrounds.

5.4 Liquid Scintillation Counter

Sample Changer Cleaning – The sample changer assembly should be vacuumed or wiped out periodically to minimize the accumulation of dust in the instrument.

5.5 Lucas Cell Counters

PMT Cleaning – On a weekly basis, the window of the PMT shall be cleaned with glass cleaner to reduce the interference from dust accumulation.

5.6 VMS backup system

5.6.1 Software Backups – On a daily basis, the sample analysis records and any software changes on the workstation data disk are backed up to a remote server that is backed up/maintained by the CST Department.

5.6.1.1 To restore files from the backup storage locations, submit a request via the CST Help Desk.

5.6.2 System Hard Drive Backup and Crash Recovery – The operating system hard drive (typically DKA0:) shall periodically be backed up to a reserve hard drive.

5.6.2.1 Backup and restoration of the operating system hard drive shall only be done by qualified personnel.

5.6.2.2 A backup shall be made at a minimum of every 180 days, whenever significant changes are made to the software on the operating system drive, or whenever the system hard drive is replaced.

5.6.2.3 Backing up the system drive

5.6.2.3.1 Ensure that no instruments are acquiring data.

5.6.2.3.2 Verify volume label for the system drive.

5.6.2.3.3 Perform a controlled system shut down.

5.6.2.3.4 Turn off the VMS system.

5.6.2.3.5 Install a spare SCSI hard drive.

5.6.2.3.6 Boot the system with an operating system CD.

5.6.2.3.7 Select the option to execute DCL commands and procedures.

5.6.2.3.8 Mount the system drive.

5.6.2.3.9 Initialize the backup drive with the same volume name as the system drive.

5.6.2.3.10 Mount the backup drive using the /foreign qualifier.

5.6.2.3.11 Execute the following command –
backup/image 'source' 'destination', where 'source' is the system drive (usually dka0:) and 'destination' is the backup drive.

5.6.2.3.12 When the backup is complete, shut down the system, disconnect the spare drive, and restart the system.

5.6.2.3.13 Label the spare drive as '**system**' **system drive backup 'date'**, where 'system' is the name of the system and 'date' is the date the backup was done. Store this drive in a safe location.

5.6.2.4 Recovering from a system drive crash

5.6.2.4.1 If possible, perform a controlled system shut down.

5.6.2.4.2 Turn off the VMS system.

5.6.2.4.3 Replace the defective system drive with the most recent system drive backup. The backup

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drive should be set to the same SCSI id as the drive being replaced.

5.6.2.4.4 Perform a backup of the new system drive as specified in section 5.6.2.3.

5.6.2.4.5 Restart the system.

5.6.3 Data Hard Drive Backup and Crash Recovery – On a weekly basis, the sample analysis records and any software changes on the workstation data disk (typically DKA100:) are automatically synchronized to a secondary hard drive in the system (typically dka200:).

5.6.3.1 Backing up the data drive - The data drive is automatically backed up on a weekly basis. No user interaction is required for this.

5.6.3.2 Recovering from a data drive crash

5.6.3.2.1 Restoration of the data drive shall only be done by qualified personnel.

5.6.3.2.2 If possible, perform a controlled system shut down.

5.6.3.2.3 Turn off the VMS system.

5.6.3.2.4 Remove the defective data drive from the system.

5.6.3.2.5 Set the SCSI id of a spare drive to that of the secondary drive.

5.6.3.2.6 Change the SCSI id of the secondary data drive to that of the defective drive.

5.6.3.2.7 Install the spare drive as a new secondary drive.

5.6.3.2.8 Boot the VMS system and log in as system administrator.

5.6.3.2.9 Copy data from the network backup that was saved since the last synchronization to the data drive with the following command –
copy/log/modified/replace/since='dd-mmm-yyyy' dnfs3:[000000...]*.*;*
'datadrive'[*]*.*;*, where 'dd-mmm-yyyy' is the date of the last synchronization and 'datadrive' is the name of the data drive (typically dka100:).

5.6.3.2.10 Initialize the new secondary drive with the same volume name as the data drive.

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5.6.3.2.11 Log off the system administrator account and log in as the operator.

5.6.3.2.12 An immediate backup of the data drive should be performed by entering the command
@backupdatadrive.

5.7 Restoring VMS Backup access after server archive

5.7.1 Periodically, the VMS backup on the server shall be archived to tape and the data removed from the server. The Computer Services Team (CST) controls the periodicity and completion of this backup. CST personnel will inform the appropriate Group Leader or designee when this operation is performed.

5.7.2 For the backup to continue operating properly, the following must be completed on each VMS system.

5.7.2.1 Log into VMS system as system administrator.

5.7.2.2 Type **search systartup_vms.com dnfs**. This will display a line showing how the backup is mounted. The last part of the line (following "backup2/") is the backup folder for the system.

5.7.2.3 Using a PC, recreate the backup folder (all lowercase letters). Set the security privileges for the new folder to allow full access.

5.7.2.4 On the VMS system, dismount the backup drive with 'dismount dnfs3:'

5.7.2.5 Remount the drive using the exact same command found in the search from step 5.7.2.2.

5.7.2.6 Example of the above:

5.7.2.6.1 SYSMGR> search systartup_vms.com dnfs3
\$TCPIP mount dnfs3: /system/host = "backsvr"
/path="/mnt01/backup2/env_alpha"

(Using a PC create the folder
\\linuxsvr05\rad_backup\env_alpha and set permissions)

SYSMGR> dismount dnfs3:

SYSMGR> TCPIP mount dnfs3:/system/host=
"backsvr"/path= "/mnt01/backup2/env_alpha"
%TCPIP\$DNFSMOUNT-S-MOUNTED,
/mnt01/backup2/env_alpha mounted on
_DNFS3:[000000]

5.7.2.7 Log off the administrator account and return to the standard user (for bioassay alpha spec, use bio_alpha).

- 5.7.2.8 Enter the command **copy/log dka100:[000000]*.dir;**
*** dnfs3:[000000]*.dir;** and wait for it to complete.
- 5.7.2.9 Enter the command **copy/log dka100:[000000...]*.dir;**
*** dnfs3:[*]*.dir;** and wait for it to complete.
- 5.7.2.10 Edit the file last_backup.lis (last_backup.list on gamma spec.) and change the date to when the tape archive of the server was made or earlier if desired.

6.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

- 6.1 Personnel performing this analytical procedure are trained to the safe laboratory practices outlined in the Safety, Health, and Chemical Hygiene Plan, GL-LB-N-001.
- 6.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.
- 6.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.
- 6.4 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.
- 6.5 Wear cryogenic gloves whenever working with liquid nitrogen. When liquid nitrogen is dispensed in the count room, leave doors open to provide proper ventilation.

7.0 RECORDS MANAGEMENT

All raw data, calculation spreadsheets, and batch checklists are filed with the sample data and maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

8.0 REFERENCES

None

9.0 HISTORY

Revision 20: Updated backup process.

Revision 19: Replaced Weekly liquid nitrogen fill with liquid nitrogen fill in section 5.1.1.

Revision 18: Added new procedures to be used when data is archived from the server.

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**STANDARD OPERATING PROCEDURE
FOR
MANAGING STATISTICAL DATA
IN THE
RADIOCHEMISTRY LABORATORY**

(GL-RAD-I-012 REVISION 26)

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1.0 STANDARD OPERATING PROCEDURE FOR MANAGING STATISTICAL DATA IN THE RADIOCHEMISTRY LABORATORY**2.0 PURPOSE**

This procedure provides guidelines on how statistical limits utilized for the verification of instrument performance are derived and used at GEL Laboratories, LLC.

3.0 DISCUSSION

Verification of the operational performance of instrumentation is critical in the production of quality radioanalytical data. Statistical data is used to determine acceptable limits, identify trends and provide other useful information. Performance checks shall be performed using appropriate check sources and monitored with control charts or tolerance charts to ensure that the instrument is operating properly, the detector response has not significantly changed and therefore the instrument calibration has not changed. The same check source used in the preparation of the tolerance or control chart at the time of the calibration should be used in the calibration verification of the instrument.

4.0 DEFINITIONS

4.1 CPM: Counts per minute.

4.2 Detector Lockout: The result of a detector failing specific performance checks causing the detector to be removed from service.

4.3 FWHM (Full width at half maximum): A measure of the resolution of a peak width at half the maximum height of the peak.

4.4 AlphaLIMS: GEL Laboratories, LLC Laboratory Information Management System

5.0 PROCEDURES

5.1 Appendix 1 gives the instrument parameters that are monitored in the Radiochemistry Counting Laboratory. The limits for these parameters are established using one of two principal mechanisms:

5.1.1 Control Charts are established by running at least 10 replicates of the given test and then calculating the mean and standard deviation. The appropriate limits are based on the standard deviation of the data set. These established limits are static until the next statistical evaluation is performed, as determined by the Group or Team Leader.

5.1.2 Tolerance Charts are established setting a fixed upper and lower boundary level. These limits are based on well-established performance specifications and cannot be more restrictive than control chart statistical limits.

5.2 The following guidelines are used to evaluate the detectors in the laboratory for all instruments that use a database to evaluate their statistical data:

A "detector lockout" occurs if the measured value is outside the limits specified in Appendix 1. The detector will be locked out until the cause of the status is investigated and a performance check is completed within tolerance. The investigation will include review of the applicable control

and/or tolerance charts for the identification of trends. Should a trend be noted, the need for any corrective action will be discussed with the Group/Team Leader and documented in the instrument maintenance logbook. The review of out of specification parameters should include the review of the instrument performance from the previous day. In addition, a review of any data generated on the out of specification instrument, from the previous day, should be evaluated to determine if it was impacted by the out of specification parameter(s).

5.3 Gas Flow Proportional Counters

- 5.3.1 Daily Efficiency Checks— Separate alpha and beta emitting radioactive sources are counted each day the detector is used. The alpha and beta sources are counted separately until a minimum of 2,000 counts are accumulated.
- 5.3.2 Daily Background Checks - A background planchet is counted for 60 minutes each day that the detector is used. This value is used to monitor instrument contamination and is typically not used for background correction of sample analyses. An administrative cap on the allowable background is 0.3 cpm alpha and 2 cpm beta.
- 5.3.3 Weekly Environmental Backgrounds - Each week an environmental background is collected for the purpose of background correcting sample analyses. The normal weekly backgrounds are counted for 500 minutes. Once per month the weekly background is collected for 1000 minutes. The weekly background checks are included with the daily background checks in the determination of statistical limits for control charts. An administrative cap on the allowable background is 0.3 cpm alpha and 2 cpm beta.
- 5.3.4 If the instrument checks are outside the established limits list in Appendix 1, the failed check should be immediately re-run according to the steps listed below. Once checks are completed, the appropriate logout sign will be placed on the locked out detectors. The instrument can be returned to service, after being locked out, following two successful instrument checks.
- 5.3.5 Weekly Background Failure
 - 5.3.5.1 Low Background: Run the daily efficiency check
 - 5.3.5.1.1 If the efficiency check passes, approve for use.
 - 5.3.5.1.2 If the efficiency check fails, notify the Team Leader or Group Leader for further review.
 - 5.3.5.2 High Background: Notify the Team Leader or Group Leader for review.
- 5.3.6 Daily Background Failure
 - 5.3.6.1 Low Background: Run the daily efficiency check
 - 5.3.6.1.1 If the efficiency check passes, approve for use.

5.3.6.1.2 If the efficiency check fails, notify the Team Leader or Group Leader for further review.

5.3.6.2 High Background: Recount the daily background check

5.3.6.2.1 If the 2nd count fails, notify Team Leader or Group Leader for further review.

5.3.6.2.2 If the 2nd count passes, count a 3rd time to determine the validity of the second count.

5.3.6.2.2.1 If the 3rd count passes, approve for use.

5.3.6.2.2.2 If the 3rd count fails, notify Team Leader or Group Leader for further review.

5.3.7 Daily Efficiency Failure

5.3.7.1 Low or High Efficiency-Ensure that the correct daily check source is in use and positioned correctly in the detector. Re-run the efficiency check.

5.3.7.1.1 If the 2nd count fails, notify Team Leader or Group Leader for further review.

5.3.7.1.2 If the 2nd count passes, count a 3rd time to determine the validity of the 2nd count.

5.3.7.1.2.1 If the 3rd count passes, approve for use.

5.3.7.1.2.2 If the 3rd count fails, notify Team Leader or Group Leader for further review.

5.4 Liquid Scintillation Counters

5.4.1 Daily instrument checks - A sealed, unquenched Tritium (H-3), Carbon-14 (C-14) and background vial are counted each day to verify instrument performance. The radioactive source is counted for a minimum of 20,000 counts, and the background is counted twice daily for a minimum of 30 minutes each instance.

NOTE: These background values are used to monitor instrument contamination and are not used for background correction of sample analyses.

5.4.2 If the instrument checks are outside the established limits list in Appendix 1, the failed check should be immediately re-run according to the steps listed below. Once the checks are completed, the appropriate logout sign will be placed on the locked out detectors. The instrument can be returned to service, after being locked out, following two successful instrument checks.

5.4.3 Daily Background Failure

5.4.3.1 Low background: Run the daily efficiency check

5.4.3.1.1 If the efficiency passes, approve for use.

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5.4.3.1.2 If the efficiency check fails, notify the Team Leader or Group Leader for further review.

5.4.3.2 High Background: Recount the daily background check

5.4.3.2.1 If the 2nd count fails, notify the Team Leader or Group Leader for further review.

5.4.3.2.2 If the 2nd count passes, count a 3rd time to determine the validity of the 2nd count.

5.4.3.2.2.1 If the 3rd count passes, approve for use.

5.4.3.2.2.2 If the 3rd count fails, notify Team Leader or Group Leader for further review.

5.4.4 Daily Efficiency Failure

5.4.4.1 Low or High Efficiency- Ensure that the correct daily check source is in use and positioned correctly in the rack. Re-run the efficiency check.

5.4.4.1.1 If the 2nd count fails, notify Team Leader or Group Leader for further review.

5.4.4.1.2 If the 2nd count passes, count a 3rd time to determine the validity of the 2nd count.

5.4.4.1.2.1 If the 3rd count passes, approve for use.

5.4.4.1.2.2 If the 3rd count fails, notify Team Leader or Group Leader for further review.

5.5 Alpha Spectrometers

5.5.1 Daily Pulser Checks - A daily pulser check is run on each detector that is to be used for analyzing samples that day. The pulser is an electronic signal typically set at 5 MeV. If the instrument checks are outside the established limits listed in Appendix 1, the failed check should be immediately rerun. If the check fails a second time the instrument is locked out of service and the cause investigated. The instrument status board will be updated to reflect the lockout condition and a logbook entry will be made. The instrument can be returned to service following two successful instrument checks.

5.5.2 Weekly Backgrounds - Once per week a background of at least 1000 minutes is counted on each detector. The background spectra is processed with each individual isotopic region of interest. If the instrument background checks are outside the established limits list in Appendix 1 for any isotope, the instrument status board will be updated to reflect the lockout condition for that particular isotope/detector. The lockout condition can be cleared following two successful background checks.

5.5.3 Monthly Calibrations - Monthly, a calibrated mixed alpha source consisting of Gd-148, Cm-244 and Np-237 is counted on each detector. The standards are used to calibrate for both energy and efficiency and to monitor the instrument performance. The analysis data is compared to the limits in Appendix 1. In the event an Alpha Spectrometer parameter is determined to be outside of the control boundaries, the status board shall be updated to reflect the "out of service" condition for that instrument and a logbook entry shall be made. The condition should be investigated and the instrument may be returned to service following two successful instrument checks and/or calibrations.

5.6 Gamma Spectrometers

5.6.1 Weekly Background Count - Once per week a background of at least 1000 minutes is counted on each detector. This count is used to subtract the background counts from sample counts. Occasionally a geometry-specific background count may be counted as needed. This is done in addition to the weekly background count and may be used for any sample counted within 1 calendar month of it. Below are the guidelines for handling any failures of the weekly background:

5.6.1.1 High background-Notify the Team Leader or Group Leader for further review.

5.6.1.2 Low background-Run the daily efficiency check.

5.6.1.2.1 If the calibration check passes, approve for use.

5.6.1.2.2 If the calibration check fails, notify the Team Leader or Group Leader for further review.

5.6.2 Daily Background Check – A 15 minute quality control count performed at least once per day a detector is in operation. This count is used to monitor contamination and is not used for background subtraction. Below are the guidelines for handling any failures.

5.6.2.1 High background-recount the daily background check

5.6.2.1.1 If the 2nd count fails, notify Team Leader or Group Leader for further review.

5.6.2.1.2 If the 2nd count passes, count a 3rd time to ensure the validity of the 2nd count.

5.6.2.1.2.1 If the 3rd count passes, approve for use.

5.6.2.1.2.2 If the 3rd count fails, notify the Team Leader or Group Leader for further review.

5.6.2.2 Low background-Run the daily efficiency check

5.6.2.2.1 If the efficiency check passes, approve for use.

5.6.2.2.2 If the efficiency check fails, notify the Team Leader or Group Leader for further review.

5.6.3 Daily Efficiency Checks – A mixed gamma standard is counted daily or prior to sample analyses. Three lines are chosen and monitored typically Am-241 at 59.5 keV, Cs-137 at 661.6 keV, and Co-60 at 1332.5 keV. For low energy detectors, such as X-ray detectors, different nuclides are chosen. The parameters monitored can be found in appendix 1 of this procedure. This is used to monitor instrument performance.

5.6.3.1 Decay corrected activity – Ensure the daily check source was positioned properly on the detector and the detector itself is positioned properly and recount.

5.6.3.1.1 If the 2nd count fails, notify the Team Leader or Group Leader for further review.

5.6.3.1.2 If the 2nd count passes, recount a 3rd time to ensure the validity of the 2nd count.

5.6.3.1.2.1 If the 3rd passes, approve for use.

5.6.3.1.2.2 If the 3rd count fails, notify the Team Leader or Group Leader for further review.

5.6.3.2 Peak FWHM – Ensure the detector is positioned properly and recount.

5.6.3.2.1 If the 2nd count fails, notify the Team Leader or Group Leader for further review.

5.6.3.2.2 If the 2nd count passes, count a 3rd time to ensure the validity of the 2nd count.

5.6.3.2.2.1 If the 3rd count fails, notify the Team Leader or Group Leader for further review.

5.6.3.2.2.2 If the 3rd count passes, approve for use.

5.6.3.3 Peak energy – Ensure the daily check source and detector are positioned correctly and re-analyze the efficiency check.

5.6.3.3.1 If the 2nd count fails, notify the Team Leader or Group Leader for further review.

5.6.3.3.2 If the 2nd count passes, recount a third time to confirm the validity of the 2nd count.

5.6.3.3.2.1 If the 3rd count fails, notify the Team Leader or Group Leader for further review.

5.6.3.3.2.2 If the 3rd count passes, approve for use.

5.7 Lucas Cells

5.7.1 Daily Efficiency Check - Each day a daily check is counted for 1 minute on each detector that is to be used for analysis. The standard is a nominal source standard of Radium-226 sealed in a Lucas cell. If the instrument checks are outside the established limits list in Appendix 1, the failed check should be immediately re-run according to the steps listed below. Once the checks are completed, the appropriate logout sign will be placed on the locked out detectors and a logbook entry made. The instrument can be returned to service after being locked out following two successful instrument checks.

5.7.1.1 Daily Efficiency Failure

5.7.1.1.1 Low or High Efficiency – ensure that the correct daily check source is in use and positioned correctly. Re-run the efficiency check.

5.7.1.1.2 If the 2nd count fails, notify Team Leader or Group Leader for further review.

5.7.1.1.3 If the 2nd count passes, count a 3rd time to determine the validity of the 2nd count.

5.7.1.1.3.1 If the 3rd count passes, approve for use.

5.7.1.1.3.2 If the 3rd count fails, notify Team Leader or Group Leader for further review.

6.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

There are no specific safety requirements associated with the activity described in this procedure. Refer to the Safety, Health and Chemical Hygiene Plan (GL-LB-N-001) for basic safety and health information.

7.0 RECORDS MANAGEMENT

7.1 Each analysis that is performed on the instrument is documented in the run log in accordance with GL-LB-E-009 for Run Logs.

7.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data and maintained as quality records.

8.0 REFERENCES

8.1 Chieco, N.A, Bogen, D.C., Knutson, E.O. Environmental Measurements Laboratory (EML) Procedures Manual. 28th Edition, Volume 1. US Department of Energy February 1997.

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- 8.4 ANSI N42.15 "Check Sources for and Verification of Liquid Scintillation Counting Systems" September 1997.
- 8.5 ANSI N42.14 "Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides", May 1999.
- 8.6 ANSI N42.25 "Calibration and Usage of Alpha/Beta Proportional Counters, January 1997.
- 8.7 Dept. of Defense (DoD) , Dept. of Energy (DoE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.0, DoE QSAS Version 3.0, July, 2013.

9.0 HISTORY

Revision 26: Step 5.3.1 changed from 50,000 counts to 2,000 counts per DoD/DoE QSM Table 18.

Revision 25: Updated section on weekly background count.

Revision 24: Replaced "Less than 3.0 keV(tolerance)" with "3 sigma" for Gamma spectrometer limit in appendix 1.

Revision 23: Updated section 5.6.3 to conform to the gamma instrument operation SOP.

Revision 22: Technical clarification made to section 5.2 for client specific requirements.

Revision 21: Updates made to clarify the process of handling QC failures for Gamma Spec and Alpha Spec instrumentation.

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APPENDIX 1

| Instrument | Parameter | Limit |
|-----------------------|---|---|
| Proportional counter | Daily Alpha Source check | 3 Sigma |
| Proportional counter | Daily Beta Source Check | 3 Sigma |
| Proportional counter | Daily alpha and beta crosstalk | 3 Sigma |
| Proportional counter | Daily Background check | 3 Sigma (0.3 cpm alpha max., 2.0 cpm beta max.) |
| Proportional counter | Weekly Background check | 3 Sigma (0.3 cpm alpha max., 2.0 cpm beta max.) |
| Scintillation counter | Daily source check (H-3 and C-14) | 3 Sigma |
| Scintillation counter | Daily background check | 3 Sigma |
| Gamma spectrometer | Daily peak energy (using 3 energy lines) | Boundary +/- 2 keV (tolerance) |
| Gamma spectrometer | Daily peak FWHM (using 3 energy lines) | 3 Sigma |
| Gamma spectrometer | Daily decay-corrected activity (using 3 energy lines) | 3 Sigma |
| Gamma spectrometer | Daily background check-total spectrum counts | 3 Sigma |
| Gamma spectrometer | Weekly background count-total spectrum counts | 3 Sigma |
| Lucas cells | Daily Source Check | 3 Sigma (tolerance) |
| Lucas cells | Daily Background Check | < 0.267 cpm (tolerance) |

| Instrument | Parameter | Standard Limit (See Note 1) | Initial Limit (See Note 2) |
|--------------------|--|---------------------------------------|--|
| Alpha spectrometer | Daily pulser check FWHM | Boundary (1-35 keV) | |
| Alpha spectrometer | Daily pulser check peak centroid | Boundary (± 15 channels of mean) | Boundary (± 15 channels of initial average) |
| Alpha spectrometer | Daily pulser check peak energy | Boundary (± 50 keV of mean) | Boundary (± 50 keV of initial average) |
| Alpha spectrometer | Daily pulser check count rate | Boundary ($\pm 3\%$ of mean) | Boundary ($\pm 3\%$ of initial average) |
| Alpha spectrometer | Weekly Background check by isotopic region | Boundary (0.050 cpm) per region | |
| Alpha spectrometer | Monthly Cal. Energy Calibration Offset | Boundary (2300-2450 keV) | |
| Alpha spectrometer | Monthly Cal. Energy Calibration Slope | Boundary (4.7-5.3 keV/channel) | |
| Alpha spectrometer | Monthly Cal. Constant FWHM | Boundary (3-25 channels) | |

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| Alpha spectrometer | Monthly Cal. Average Efficiency | 3 Sigma std. deviation | Boundary (± 0.1 of initial average) |
| Alpha spectrometer | Monthly Cal. Peak Energy of 3 isotopes | Boundary (± 40 keV of actual energy) | |
| Alpha spectrometer | Monthly Cal. Peak Resolution of 3 isotopes | Boundary (5-100 keV) | |
| Alpha spectrometer | Monthly Cal. Peak Area of 3 isotopes | Boundary (minimum 10,000 counts) | |
| Alpha spectrometer | Monthly Cal. Peak Activity of 3 isotopes | 3 Sigma std. deviation | Boundary ($\pm 1\%$ of initial average) |

Note 1: Standard Limits are based on 3 Sigma and/or Mean and will be calculated on 20 reference points after an initial setup or repair/adjustment is made.

Note 2: Initial Limit is used when an instrument is initially set up or repaired/adjusted. These limits tend to have tighter boundaries than those used for 3 Sigma Standard Limit. Once 20 reference points are collected, the Standard limit will be calculated. Initial Averages are determined from the first 2 reference points.

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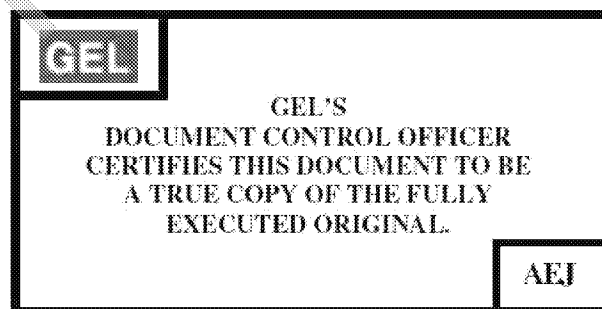
STANDARD OPERATING PROCEDURE FOR

MULTI-DETECTOR COUNTER OPERATING INSTRUCTIONS

(GL-RAD-I-016 REVISION 10)

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1.0 STANDARD OPERATING PROCEDURE FOR MULTI-DETECTOR COUNTER OPERATING INSTRUCTIONS**2.0 METHOD OBJECTIVE, PURPOSE, CODE, AND SUMMARY (IDENTIFICATION OF TEST METHOD)**

This method describes the operation of the Multi-Detector Counting System for routine sample analysis. The operation includes access to the computer operating system, calibration, performance checks, initiating data collection, and printing data plots and reports.

3.0 APPLICABLE MATRIX OR MATRICES

This procedure is applicable to all matrices.

4.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

This procedure is not specific to one particular method. For method scope, applicability, or detection limit refer to the method specific analytical standard operating procedure.

5.0 METHOD VARIATIONS

Not Applicable.

6.0 DEFINITIONS

- 6.1 Check Source: A radioactive source, not necessarily calibrated, that is used to confirm the satisfactory operation of the instrument.
- 6.2 Crosstalk: The detection of alpha events in the beta channel or the detection of beta events in the alpha channel during simultaneous counting.
- 6.3 Efficiency: The percent of decay events from a standard source that are seen and measured by a detector.
- 6.4 Proportional Counter: A gas filled radiation counter tube operated in the range of high voltage in which the total charge collected for each ionizing event is proportional to the number of ion pairs formed in the tube by the initial event.
- 6.5 Self Absorption: Absorption of radioactive emissions by the solids contained on the counting planchet, thereby preventing the emission from reaching the detector.
- 6.6 Simultaneous Counting: The measurement of both gross alpha and gross beta activity at the same time.

7.0 INTERFERENCES/LIMITATIONS

For analyses requiring isotope specific analyses (i.e. Cl-36, Sr-90), chemical separations are performed during sample preparation to remove unwanted counting interferences.

8.0 SAFETY PRECAUTIONS AND WARNING

There are no specific safety requirements associated with the activity described in this procedure. Refer to GL-LB-N-001, the Safety, Health, and Chemical Hygiene Plan, for basic safety and health information.

9.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

- 9.1 Protean Multi-detector Gross Alpha/Beta Counting System.

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- 9.2 Printer
- 9.3 Gas Regulator, recommended a dual stage regulator
- 9.4 Radioactive Check Source, containing an alpha and/or beta emitting isotope(s). The actual isotope is dependent on the radionuclide to be measured. Normally a Sr-90/Y-90 or Th-230 source is used.
- 9.5 Traceable Calibration Standard: NIST traceable standard based upon the isotope to be measured
- 9.6 P-10 Gas (90% Argon/10% Methane)

10.0 REAGENTS AND STANDARDS

Not Applicable.

11.0 SAMPLE HANDLING AND PRESERVATION

Not Applicable.

12.0 SAMPLE PREPARATION

Refer to appropriate sample preparation procedure.

13.0 QUALITY CONTROL SAMPLES

Refer to GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

14.0 INSTRUMENTATION CALIBRATION, STANDARDIZATION, AND PERFORMANCE

- 14.1 Calibration of the instrument shall be performed at initial installation. The calibration will be reestablished and/or reverified on an annual frequency, after changing detectors, following system maintenance that may affect the electronics, or when a problem is suspected. The instrument's window settings should not be changed after the initial set up. If the window settings are altered, all calibrations for the instrument must be renewed.

NOTE: For Drinking Water Compliance Monitoring-If a gas proportional counter is moved, serviced, or had an interruption in either gas flow or electrical power, the plateau voltage for both alpha and beta is verified, its crosstalk factors remeasured, and the solids absorption curves for each analyte reverified or regenerated prior to measuring any compliance monitoring samples.

NOTE: Expiration dates will match the last day of the month in which the calibration data was acquired.

NOTE: After initial Plateau generation, the regeneration of a Plateau to determine the Operating Voltage is not required, unless indicated by instrument response or performance as determined through daily performance checks, control charts, and calibration verifications (ANSI N42.25). If a generation of a Plateau is not required, proceed to step 14.2 of this calibration section.

- 14.1.1 Determining the operating voltage:

The operating voltage of the instrument is determined by performing a plateau. The plateau is performed by counting a Sr-90 source long

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enough to achieve 10,000 counts. The source is repeatedly counted as the voltage of the instrument is increased.

14.1.2 Performing a Plateau

- 14.1.2.1 Load a Sr-90 source into each one of the instruments that is to be calibrated.
- 14.1.2.2 Click on the appropriate instrument box on the instrument software. (i.e. To start a plateau for detectors 1A, 1B, 1C, 1D click on the box labeled instrument 1)
- 14.1.2.3 Select "instrument" from the software's toolbar, and choose bias selection.
- 14.1.2.4 In the pop-up window, click on the start button to run the plateau. Once the plateau has been completed, the data may be printed and graphed. The optimum operation voltage for beta counting is approximately 30-150 volts above the knee of the curve. The knee is determined by drawing straight lines along the rising slope and the plateau portions of the curve; the knee is the point where these two lines intersect. The operating voltage is typically the first point on the plateau (flat portion of the curve). The plateau length should be at least 200 volts. The % slope per 100 volts should be <10% for distributed sources. This will yield good efficiency with the lowest background.

14.2 Source preparation for the crosstalk and efficiency determinations can be located in each isotopic SOP as each calibration is matrix/isotope specific.

14.3 Crosstalk determination:

Crosstalk determination is the measurement of alpha events in the beta channel or beta events in the alpha channel. Crosstalk is determined on an annual basis, usually coinciding with the gross alpha/beta calibration. These crosstalk values are used in all calibrations for the instrument.

14.3.1 Determining alpha crosstalk

Alpha crosstalk is the measurement of alpha events in the beta channel. The value is determined by counting a set of pure alpha emitting sources (Po-210), which should increase in mass similarly to calibration efficiency sources, long enough to achieve 10,000 total alpha counts each. The alpha crosstalk value is calculated by dividing the number of resulting beta counts by the number of resulting alpha counts. Source mass vs. %crosstalk should be plotted, and the coefficients of the curve should be used in calculating alpha crosstalk for all samples.

NOTE: For Drinking Water Crosstalk determination, Th-230 will be used to determine crosstalk values. The crosstalk values will be optimized using a Po-210 mass attenuation curve.

14.3.2 Determining beta crosstalk

Beta crosstalk is the measurement of beta events in the alpha channel. A set of Sr-90 sources should be counted in each detector, long enough to achieve 10,000 total beta counts. Beta crosstalk should be calculated for each source by dividing total alpha counts by total beta counts. An average of all of the sources crosstalk values should be calculated for each detector, and this average value will represent that detector's beta crosstalk.

14.4 Efficiency determination

An efficiency for each detector is determined per method. For Ra-228 in all matrices except drinking water, an average efficiency is determined for each detector. This is accomplished by counting a set of at least 4 identical sources in each detector long enough to achieve 10,000 total beta counts. Efficiency is calculated as cpm/dpm and an average of the efficiencies for each detector is determined. For all other methods counted on the instrument, a set of sources that increase in mass, is counted long enough to achieve 10,000 total counts for each source in the respective channel. Efficiency (cpm/dpm) is determined and plotted against the mass of the source. The coefficients of the plotted curve are used to determine efficiency per sample, corresponding to the sample's mass.

NOTE: For the above generated efficiency curves, each data point should be within 10% of the calculated value of the curve. If any data points exceed the acceptance criteria, the GL should be consulted for evaluation and possible exclusion of the outlier.

14.5 Efficiency verification

The determined efficiency of each detector for each calibration must be verified with an independent NIST traceable source prior to using the newly established efficiencies. The result of each verification source when calculated with the new efficiency must be $\pm 25\%$ of the known value.

14.6 Weekly Checks

The instrument background is determined weekly using the same procedure as for the Daily Background Check (below) except it is counted for 500 minutes. Monthly, one of the weekly background counts will be extended to 1000 minutes. This will normally be done during the first week of a new month.

14.7 Daily Checks

14.7.1 Before counting samples, ensure that the daily QA and background checks have been completed and entered into AlphaLIMS.

- 14.7.1.1 Background Check: Confirm P-10 gas to instrument is between 0.1-0.3 (scfh). If not, contact group leader. Place a contamination free planchet into each detector holder. Using the mouse, double click the appropriate detector to begin its background count. A new window will appear. In that window, select "source log." From the drop down menu under sample id, highlight BKG. Enter 60 minutes in the field supplied for count time, and click the start button. Repeat this until all detector backgrounds are started.
- 14.7.1.2 Daily Efficiency Check – Place the prepared check sources in the appropriate holders. Follow the steps for starting a background count, except choose EFFA (for the alpha check source) or EFFB (for the beta check source). Count each assigned source (alpha and beta) for 5 minutes in each detector.
- 14.7.2 Uploading QA Data
 - 14.7.2.1 Open Microsoft Excel. "Rad Menus" file should open automatically.
 - 14.7.2.2 Click "GFPC Dailies" button.
 - 14.7.2.3 Enter today's date into the "Enter Date for Daily Report" field. Data will be pulled for all GFPC instruments. This may take a few moments.
 - 14.7.2.4 Enter today's date into the "Enter date to upload" field. Click "ok."
 - 14.7.2.5 Print daily and scan daily sheet or sign electronically. Refer to GL-RAD-I-012, Managing Statistical Data in the Radiochemistry Laboratory, for guidance on locking out detectors.

15.0 PROCEDURES

15.1 Startup

The Multi-Detector is left in a running condition. The software program may be closed or left open. Start the Protean software by opening the PIC MDS Control Panel.

15.2 Starting a Count

- 15.2.1 Confirm P-10 gas to instrument is between 0.1-0.3 (scfh). If not, contact group leader.
- 15.2.2 Insert samples into counting unit and mark detector IDs in the appropriate area of the que sheet.

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- 15.2.3 From the tool bar at the top of the monitor, click “log” and from the drop down menu select batch. Click the + button, and enter the batch #. Click the + button again.
- 15.2.4 From the tool bar at the top of the monitor, click “log” and from the drop down menu select sample. Click the + button and enter the sample ID. From the drop down menu, select the batch #. Repeat this for all loaded samples. When done, close the log window.
- 15.2.5 Double click on the detector ID to be started. Select the sample ID from the drop down menu, and enter the count time. Click start.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-010, Counting Room Instrument Maintenance and Performance Checks.

17.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Refer to GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

18.0 POLLUTION/CONTAMINATION

Not Applicable

19.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Refer to GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

20.0 CORRECTIVE ACTIONS FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

Corrective action for out-of-control data might require instrument maintenance, reanalysis, using a new spike mix, or a more complex set of actions. When troubleshooting measures fail to bring an analytical process or data into control, a Data Exception Report (DER) and/or corrective action should be initiated in accordance with GL-QS-E-004 for Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items, and/or GL-QS-E-002 for Conducting Corrective/Preventive Action and Identifying Opportunities for Improvement.

21.0 CONTINGENCIES FOR HANDLING THESE SITUATIONS

Troubleshooting the instrument is a function of analyst experience. In-house service is obtained from GEL’s group leader or other qualified personnel. If vendor assistance is needed, then the appropriate vendor is contacted. Maintenance logbooks are kept for each instrument and contain entries for both routine and non-routine maintenance procedures.

22.0 RECORDS MANAGEMENT

- 22.1 Each sample analysis that is performed is documented in the instrument run log in accordance with GL-LB-E-009 for Run Logs.
- 22.2 All raw data printouts, calculation spreadsheets, and batch checklists are filed with the sample data for archive in accordance with GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

16-Aug-2018

Multi-Detector Counter Operating Instructions

SOP Effective September 2001
Revision 10 Effective April 2015

GL-RAD-I-016 Rev 10
Page 9 of 9

22.3 Instrument maintenance is recorded in accordance with GL-LB-E-008, Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.

23.0 LABORATORY WASTE HANDLING AND DISPOSAL

Refer to GL-LB-G-001, GEL's Laboratory Waste Management Plan.

24.0 REFERENCES

24.1 Protean Instruments Multi-detector System Operation Manual.

24.2 ANSI N42.26-1997 Calibration and Usage of Alpha/Beta Proportional Counters.
NOTE: GEL incorporates this reference with the exception of section 6.5 for Effectiveness of the Guard detector which is not incorporated for use. The efficiency sources used in the calibration process typically achieve 10,000 gross counts and the sources used in the cross talk and verification process typically achieve 5,000 gross counts.

24.3 DOE Quality Systems for Analytical Services

25.0 HISTORY

Revision 5: Updated procedures for uploading QC data from GFPC instruments.

Revision 6: Updated section on instrument calibration for clarification.

Revision 7: Calibration clarification.

Revision 8: Updated for drinking water compliance monitoring to comply with DHEC certification extension requirements.

Revision 9: Updated to include drinking water crosstalk determination using Th-230 and Po-210.

Revision 10: Added NOTE to section 24.2.

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Attachment 4

Laboratory Certifications



SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

GEL LABORATORIES, LLC
2040 Savage Road
Charleston, SC 29414
Robert L. Pullano Phone: (843) 556-8171
rlp@gel.com

ENVIRONMENTAL

Valid To: June 30, 2019

Certificate Number: 2567.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.1 of the DoD Quality Systems Manual for Environmental Laboratories), accreditation is granted to this laboratory to perform the following radiochemical tests in various matrices, including soils, drinking water, wastewater, groundwater, fiber air filters, vegetation, animal tissues, milk and construction debris:

| <u>Test(s)</u> | <u>Preparation SOP(s)</u> | <u>Analytical SOP(s)</u> |
|--|--|-------------------------------|
| <u>Alpha Spectrometry:</u> Alpha: Am-241, Am-243, Cf-252, Cm-242, Cm-243/244, Cm-245/246, Np-237, Po-208, Po-209, Po-210, Pu-236, Pu-238, Pu-239/240, Pu-241, Pu-242, Pu-244, Ra-224, Ra-226, Th-228, Th-229, Th-230, Th-232, U-232, U-233/234, U-235/236, U-238 | GL-RAD-A-011, GL-RAD-A-016, GL-RAD-A-032, GL-RAD-A-035, GL-RAD-A-036, GL-RAD-A-038, GL-RAD-A-046 | GL-RAD-I-009, GL-RAD-I-015 |
| <u>Radon Emanation:</u> Ra-226 | GL-RAD-A-008, GL-RAD-A-028 | GL-RAD-I-007 |
| <u>Gamma Spectrometry:</u> Gamma: 46 to 1836 keV, I-129, I-131, Ni-59 | GL-RAD-A-006, GL-RAD-A-013, GL-RAD-A-022 | GL-RAD-I-001 |
| <u>Kinetic Phosphorescence Analyzer:</u> Total Uranium | GL-RAD-A-023 | GL-RAD-B-018 |

| <u>Test(s)</u> | <u>Preparation SOP(s)</u> | <u>Analytical SOP(s)</u> |
|---|---|---|
| <u>Gas Flow Proportional Counting:</u> Alpha: Total Radium 48 Hour Gross Alpha Gross Alpha/Gross Beta Beta: Cl-36, I-131, Pb-210, Ra-228, Sr-89, Sr-90 | GL-RAD-A-010, GL-RAD-A-044 GL-RAD-A-047 GL-RAD-A-001, GL-RAD-A-001B, GL-RAD-A-001C, GL-RAD-A-001D GL-RAD-A-004, GL-RAD-A-009, GL-RAD-A-017, GL-RAD-A-018, GL-RAD-A-029, GL-RAD-A-030, GL-RAD-A-033, GL-RAD-A-054, GL-RAD-A-058 | GL-RAD-I-006, GL-RAD-I-015, GL-RAD-I-016, GL-RAD-I-021 |
| <u>Liquid Scintillation Spectrometry:</u> Gross Alpha/Gross Beta Alpha: Rn-222 Beta: C-14, Ca-45, Fe-55, H-3, Ni-63, P-32, Pm-147, Pu-241, S-35, Se-79, Tc-99 Pyrolysis Preparation C-14, H-3 (Special Matrices) | GL-RAD-A-056 GL-RAD-A-007 GL-RAD-A-002, GL-RAD-A-003, GL-RAD-A-005, GL-RAD-A-019, GL-RAD-A-020, GL-RAD-A-022, GL-RAD-A-031, GL-RAD-A-035, GL-RAD-A-040, GL-RAD-A-048, GL-RAD-A-049, GL-RAD-A-050, GL-RAD-A-059 GL-RAD-A-067 | GL-RAD-I-004, GL-RAD-I-014, GL-RAD-I-017 |
| <u>ICP-MS:</u> Uranium Isotopes, Tc-99 | GL-RAD-A-005, GL-RAD-A-055 | GL-RAD-B-034 |



Additionally, In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.1 of the DoD Quality Systems Manual for Environmental Laboratories), accreditation is granted to this laboratory to perform recognized EPA, Standard Methods for the Examination of Water and Wastewater, ASTM, California and Connecticut test methods using the following testing technologies and in the analyte categories identified below:

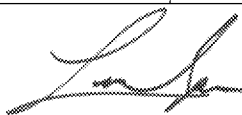
Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Methylene Blue Active Substances, Misc.-Electronic Probes (pH, O₂), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), IR Spectrometry, Titrimetry, Total Organic Carbon, Total Organic Halide, Turbidity, Liquid Chromatography/Mass Spectrometer/Mass Spectrometer and Various Radiochemistry Techniques

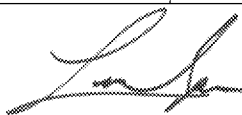
| <u>Parameter/Analyte</u> | <u>Potable Water</u> | <u>Aqueous Film Forming Foams (AFFF)</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|---|----------------------|--|--|--|
| <u>Per-and Polyfluoroalkyl Substances (PFAS)</u> | | | | |
| Fluorotelomer sulfonate 4:2 (4:2 FTS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Fluorotelomer sulfonate 6:2 (6:2 FTS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Fluorotelomer sulfonate 8:2 (8:2 FTS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorobutanesulfonate (PFBS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorobutyric acid (PFBA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |



| <u>Parameter/Analyte</u> | <u>Potable Water</u> | <u>Aqueous Film Forming Foams (AFFF)</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|-----------------------------------|----------------------|--|--|--|
| Perfluorodecanesulfonate (PFDS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorodecanoic acid (PFDA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorododecanoic acid (PFDoA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluoroheptanesulfonate (PFHpS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluoroheptanoic acid (PFHpA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorohexanesulfonate (PFHxS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorohexanoic acid (PFHxA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorononane sulfonate (PFNS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |



| <u>Parameter/Analyte</u> | <u>Potable Water</u> | <u>Aqueous Film Forming Foams (AFFF)</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--------------------------------------|----------------------|--|--|--|
| Perfluorononanoic acid (PFNA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorooctanesulfonamide (PFOSA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorooctanoic acid (PFOA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluoropentanoic acid (PFPeA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorotetradecanoic acid (PFTeDA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluorotridecanoic acid (PFTrDA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluoroundecanoic acid (PFUdA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Perfluoropentanesulfonate (PFPeS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |



| <u>Parameter/Analyte</u> | <u>Potable Water</u> | <u>Aqueous Film Forming Foams (AFFF)</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--|----------------------|--|--|--|
| Perfluorooctanesulfonic acid (PFOS) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| N-ethyl perfluorooctanesulfonamidoacetic acid (N-EtFOSAA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| N-methyl perfluorooctanesulfonamidoacetic acid (N-MeFOSAA) | EPA 537 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 | EPA 537 Mod, PFAS by LCMSMS Compliant with QSM 5.1 Table B-15 |
| Propanoic acid (PFPrOPrA) – GenX | EPA 537 | EPA 537 Mod* | EPA 537 Mod* | ----- |

** Non DoD work*

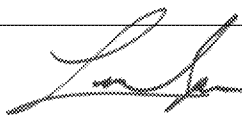
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--------------------------|--|--|
| <u>Metals</u> | | |
| Aluminum | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Antimony | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B ² |
| Arsenic | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Barium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Beryllium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Bismuth | EPA 200.8/6020 EPA 6020/6020A/6020B | EPA 6020/6020A/6020B |
| Boron | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |



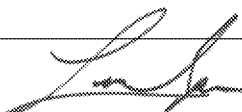
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--------------------------|--|---|
| Cadmium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Calcium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Chromium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Cobalt | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Copper | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Hafnium | EPA 200.8 EPA 6020/6020A/6020B | EPA 6020/6020A/6020B |
| Iron | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Lead | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Lithium | EPA 200.8 EPA 6020/6020A/6020B | EPA 6020/6020A/6020B |
| Magnesium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Manganese | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Mercury | EPA 245.1/245.2 EPA 7470/7470A EPA 1631E | EPA 7470/7470A EPA 7471A/7471B |
| Molybdenum | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Nickel | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Phosphorous | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Potassium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Rhenium | EPA 200.8 EPA 6020/6020A/6020B | EPA 6020/6020A/6020B |
| Rhodium | EPA 200.8 EPA 6020A/6020B | EPA 6020/6020A/6020B |



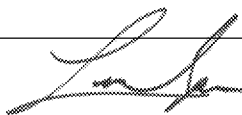
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|-----------------------------------|---|--|
| Selenium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Silicon ¹ | EPA 200.7 EPA 6010B/6010C/6010D | EPA 6010B/6010C/6010D |
| Silica as SiO ₂ | EPA 200.7 EPA 6010B/6010C/6010D | EPA 6010B/6010C/6010D |
| Silver | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B ² |
| Sodium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Strontium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Sulfur | EPA 200.7 EPA 6010B/6010C/6010D | EPA 6010B/6010C/6010D |
| Tantalum | EPA 6020/6020A/6020B | EPA 6020/6020A/6020B |
| Thallium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Thorium | EPA 200.8 EPA 6020/6020A/6020B | EPA 6020/6020A/6020B |
| Tin | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B ² |
| Titanium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Tungsten | EPA 200.8 EPA 6020/6020A/6020B | EPA 6020/6020A/6020B |
| Uranium | EPA 200.8 EPA 6020/6020A/6020B ASTM D5174-02/97 DOE U-02 | EPA 6020/6020A/6020B DOE U-02 |
| Isotopic Uranium | EPA 200.8 EPA 6020A/6020B | EPA 6020A/6020B |
| Vanadium | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Zinc | EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B | EPA 6010B/6010C/6010D EPA 6020/6020A/6020B |
| Zirconium | EPA 200.8 EPA 6020A/6020B | EPA 6020A/6020B |
| <u>General Chemistry</u> | | |
| Acidity | EPA 305.1 SM 2310B | ----- |
| Adsorbable Organic Halogens (AOX) | EPA 1650 | ----- |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|---|---|---|
| Alkalinity | EPA 310.1 SM 2320B | ----- |
| Ammonable Cyanide | EPA 9012A/9012B EPA 335.1 SM 4500-CN ⁻ G | EPA 9012A/9012B |
| Ammonia Nitrogen (and distillation) | EPA 350.1 SM 4500NH ₃ B/H | EPA 350.1 Modified |
| Biochemical Oxygen Demand (BOD) | EPA 405.1 SM 5210 B | ----- |
| Bromide | EPA 300.0 EPA 9056A | EPA 9056A ³ |
| Carbon Dioxide (Total and Free by calculation) | SM 4500-CO ₂ D | ----- |
| Carbonaceous BOD (CBOD) | EPA 405.1 SM 5210B | ----- |
| Chemical Oxygen Demand (COD) | EPA 410.4 SM 5220D | ----- |
| Chloride | EPA 300.0 EPA 9056A | EPA 9056A ³ |
| Chlorine (residual) | EPA 330.5 SM 4500-Cl G | ----- |
| Chromium VI | EPA 7196A SM 3500-Cr B | EPA 7196A |
| Color | EPA 110.2 SM 2120B | ----- |
| Corrosivity toward Steel | ----- | EPA 1110/1110A |
| Cyanide | EPA 335.4 EPA 9012A/9012B SM4500-CN ⁻ E/G | EPA 9012A/9012B |
| Density | ----- | ASTM D5057 |
| Extractable Organic Halides (EOX) | ----- | EPA 9023 |
| Fluoride | EPA 300.0 EPA 9056A | EPA 9056A ³ |
| Ignitability | EPA 1020A/1020B | EPA 1020A/1020B |
| Iodide | EPA 300.0 EPA 9056A | EPA 9056A |
| Hardness (by calculation/titration) | EPA 130.2 EPA 200.7/200.8 EPA 6010B/6010C/6010D EPA 6020/6020A/6020B SM 2340B/C | EPA 6010B/6010C/6010D EPA 6020A/6020B |
| Kjeldahl Nitrogen (TKN) | EPA 351.2 SM 4500N _{org} D | EPA 351.2 Modified |
| MBAS/Surfactants | EPA 425.1 SM 5540C | ----- |
| Nitrate (as N) | EPA 300.0 EPA 9056A SM4500-NO ₃ -F | EPA 9056A ³ |
| Nitrate-nitrite (as N) | EPA 300.0, 353.2 EPA 9056A SM 4500 NO ₃ -F | EPA 9056A ³ |



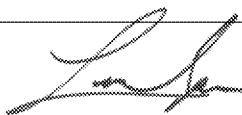
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|-------------------------------------|---|---|
| Nitrite (as N) | EPA 300.0 EPA 9056A | EPA 9056A ³ |
| Oil & Grease | EPA 1664A | EPA 1664A ² |
| Organic Nitrogen | EPA 350.1 EPA 351.2 TKN – Ammonia | ----- |
| Orthophosphate (as P) | EPA 300.0 EPA 9056A | EPA 9056A ³ |
| Oxygen, Dissolved | SM 4500O G | ----- |
| Paint Filter Liquids Test | ----- | EPA 9095B |
| Perchlorate | EPA 314.0 EPA 6850 | EPA 314.0 Modified EPA 6850 |
| pH | EPA 150.1 EPA 9040B/9040C EPA 9041A SM 4500-H ⁺ B | EPA 9040B/9040C EPA 9045C/9045D |
| Reactive Cyanide | Sec 7.3.3 SW846 | Sec 7.3.3 SW846 |
| Reactive Sulfide | Sec 7.3.4 SW846 | Sec 7.3.4 SW846 |
| Residue- Filterable (TDS) | EPA 160.1 SM 2540C | ----- |
| Residue- Nonfilterable (TSS) | EPA 160.2 SM 2540D | ----- |
| Residue- Total | EPA 160.3 SM 2540B | ----- |
| Residue- Total, fixed, and volatile | SM 2540G | ----- |
| Residue- Volatile | EPA 160.4 SM 2540E | ----- |
| Salinity | SM 2520B | ----- |
| Specific conductance | EPA 120.1 EPA 9050A/120.1 SM 2510B | ----- |
| Sulfate | EPA 300.0 EPA 9056A | EPA 9056A ³ |
| Sulfite | SM 4500-SO ₃ ²⁻ B | ----- |
| Sulfide | EPA 376.2 EPA 9030B EPA 9034 SM 4500 S ²⁻ D | EPA 9030B EPA 9034 |
| Total Nitrate-Nitrite | EPA 353.2 SM 4500-NO ₃ F | ----- |
| Total Organic Carbon (TOC) | EPA 9060/9060A SM 5310B/415.1 | EPA 9060/9060A ² |
| Total Organic Halides (TOX) | EPA 9020B | EPA 9020B ² |
| Total Petroleum Hydrocarbons | EPA 1664A | EPA 1664A |
| Total Phenolics | EPA 420.4 EPA 9066 | EPA 9066 |
| Total Phosphorous | EPA 365.4 SM 4500-P H | EPA 365.4 Modified |
| Turbidity | EPA 180.1 SM 2130B | ----- |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--|--|---|
| <u>Organic Analytes</u> | | |
| 1,2-Dibromo-3-chloropropane (DBCP) | EPA 504.1 EPA 624.1 EPA 8011 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,2 Dibromoethane (EDB) | EPA 504.1 EPA 624.1 EPA 8011 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,2,3-Trichloropropane | EPA 504.1 EPA 624.1 EPA 8011 EPA 8260B/8260C | EPA 8260B/8260C |
| <u>Purgeable Organics (Volatiles)</u> | | |
| 1,1,1,2-Tetrachloroethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,1,1-Trichloroethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,1,2,2-Tetrachloroethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,1,2-Trichloroethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,1-Dichloroethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,1-Dichloroethene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,1-Dichloropropene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,2,3-Trichlorobenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,2,3-Trichloropropane | EPA 504.1 EPA 624.1 EPA 8011 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,2,4-Trichlorobenzene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| 1,2,4-Trimethylbenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8060C |
| 1,2-Dichlorobenzene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| 1,2-Dichloroethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|----------------------------------|--|---|
| 1,2-Dichloropropane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,3,5-Trimethylbenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,3-Dichlorobenzene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| 1,3-Dichloropropane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 1,4-Dichlorobenzene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| 1,4-Dioxane | EPA 522 EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D/ | EPA 8260B/8260C EPA 8270C/8270D |
| 1-Chlorohexane | EPA 8260B | EPA 8260B |
| 2,2-Dichloropropane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 2-Butanone (Methyl Ethyl Ketone) | EPA 624.1 EPA 8015C/8015D EPA 8260B/8260C | EPA 8015C/8015D EPA 8260B/8260C |
| 2-Chloroethyl vinyl ether | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 2-Chlorotoluene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 2-Hexanone | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 2-Nitropropane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 2-Pentanone | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 4-Chlorotoluene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 4-Isopropyltoluene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| 4-Methyl-2-pentanone | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Acetone | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Acetonitrile | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Acrolein (propanol) | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Acrylonitrile | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Allyl Chloride | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|------------------------------------|--|---|
| Benzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Benzyl chloride | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Bis(2-chloro-1 methyl-ethyl) ether | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| Bromobenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Bromochloromethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Bromodichloromethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Bromoform | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Bromomethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Carbon disulfide | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Carbon tetrachloride | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Chlorobenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Chloroethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Chloroform | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Chloromethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Chloroprene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| cis-1,2-Dichloroethene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| cis-1,3-Dichloropropene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| cis-1,4-Dichloro-2-butene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Cyclohexane | EPA 8260B/8260C | EPA 8260B/8260C |
| Cyclohexanone | EPA 8260B/8260C | EPA 8260B/8260C |
| Cyclohexene | EPA 8260B | EPA 8260B |
| Dibromochloromethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Dibromomethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Dichlorodifluoromethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Diethyl ether | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--------------------------------|---|---|
| Ethyl Acetate | EPA 624.1 EPA 8015C/8015D EPA 8260B/8260C | EPA 8015C/8015D EPA 8260B/8260C |
| Ethyl Benzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Ethyl methacrylate | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Ethyl tert-butyl ether | EPA 8260B | EPA 8260B |
| Hexachlorobutadiene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| Hexane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Iodomethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Isobutyl alcohol | EPA 624.1 EPA 8015B/8015C EPA 8260B/8260C | EPA 8260B/8260C |
| Isopropyl alcohol | EPA 8260B | EPA 8260B |
| Isopropylbenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Isopropyl ether | EPA 8260B | EPA 8260B |
| m+p-Xylene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Methacrylonitrile | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Methyl acetate | EPA 8260B/8260C | EPA 8260B/8260C |
| Methyl methacrylate | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Methyl tert-amyl ether (TAME) | EPA 8260B | EPA 8260B |
| Methyl tert-butyl ether (MTBE) | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Methylcyclohexane | EPA 8260B/8260C | EPA 8260B/8260C |
| Methylene chloride | EPA 624.1 EPA 8260B/8260C | EPA 8260C |
| Naphthalene | EPA 624.1 EPA 625.1 EPA 8310 EPA 8260B/8260C EPA 8270C/8270D ⁴ | EPA 8310 EPA 8260B/8260C EPA 8270C/8270D ⁴ |
| n-Butyl alcohol | EPA 624.1 EPA 8015C/8015D EPA 8260B/8260C | EPA 8015C/8015D EPA 8260B/8260C |
| n-Butylbenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| n-Propylbenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| o-Xylene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |



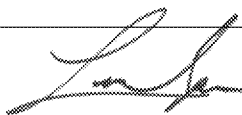
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--------------------------------------|--|---|
| Pentachloroethane | EPA 624.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| Propionitrile | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Sec-Butylbenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Styrene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| tert-Butyl Alcohol | EPA 8260B/8260C | EPA 8260B/8260C |
| tert-Butylbenzene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Tetrachloroethene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Tetrahydrofuran | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Toluene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| trans-1,2-Dichloroethene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| trans-1,3-Dichloropropene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| trans-1,4-Dichloro-2-butene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Trichloroethene | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Trichlorofluoromethane | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Trihalomethanes | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Vinyl acetate | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Vinyl chloride | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| Xylenes, total | EPA 624.1 EPA 8260B/8260C | EPA 8260B/8260C |
| <u>Semivolatile Compounds</u> | | |
| 1,2,4,5-Tetrachlorobenzene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 1,2,4-Trichlorobenzene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| 1,2-Dichlorobenzene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| 1,2-Diphenylhydrazine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|---------------------------|---|---|
| 1,3,5-Trinitrobenzene | EPA 625.1 EPA 8330A/8330B ⁵ EPA 8270C/8270D | EPA 8330A/8330B ⁵ EPA 8270C/8270D |
| 1,3-Dichlorobenzene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| 1,3-Dinitrobenzene | EPA 625.1 EPA 8330A/8330B ⁵ EPA 8270C/8270D | EPA 8330A/8330B ⁵ EPA 8270C/8270D |
| 1,4-Dichlorobenzene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| 1,4-Dioxane | EPA 522 EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| 1,4-Dinitrobenzene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 1,4-Naphthoquinone | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 1,4-Phenylenediamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 1-Methylnaphthalene | EPA 625.1 EPA 8270C/8270D ⁴ | EPA 8270C/8270D ⁴ |
| 1-Naphthylamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2,2-Dichlorobenzil | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2,3,4,6-Tetrachlorophenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2,3-Dichloroaniline | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2,4,5-Trichlorophenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2,4,6-Trichlorophenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2,4-Dichlorophenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2,4-Dimethylphenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2,4-Dinitrophenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2,4-Dinitrotoluene | EPA 625.1 EPA 8330A/8330B ⁵ EPA 8270C/8270D | EPA 8330A/8330B ⁵ EPA 8270C/8270D |
| 2,6-Dichlorophenol | EPA 625.1/8270C/8270D | EPA 8270C/8270D |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--------------------------------|--|---|
| 2,6-Dinitrotoluene | EPA 625.1 EPA 8330A/8330B ⁵ EPA 8270C/8270D | EPA 8330A/8330B ⁵ EPA 8270C/8270D |
| 2-Acetylaminofluorene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2-Butoxyethanol | EPA 8270C/8270D | EPA 8270C/8070D |
| 2-Chloronaphthalene | EPA 625.1 EPA 8270C/8270D ⁴ | EPA 8270C/8270D ⁴ |
| 2-Chlorophenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2-Ethoxyethanol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2-Methyl-4,6-Dinitrophenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2-Methylnaphthalene | EPA 625.1 EPA 8270C/8270D ⁴ | EPA 8270C/8270D |
| 2-Methylphenol (o-cresol) | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2-Naphthylamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2-Nitroaniline | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2-Nitrophenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 2-Picoline (2-Methylpyridine) | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 3,3'-Dichlorobenzidine | EPA 625.1 EPA 8270C/8270D ⁴ | EPA 8270C/8270D |
| 3,3'-Dimethylbenzidine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 3/4-Methylphenols(m/p cresols) | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 3-Methylcholanthrene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 3-Nitroaniline | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 4,4-Dichlorodiphenyl sulfone | EPA 8270C/8270D | EPA 8270C/8270D |
| 4-Aminobiphenyl | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 4-Bromophenyl phenyl ether | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 4-Chloro-3-methylphenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 4-Chloroaniline | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 4-Chlorophenyl phenyl ether | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 4-Nitroaniline | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 4-Nitrophenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|-------------------------------------|---|---|
| 5-Nitro-o-toluidine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| 7,12-Dimethylbenz(a)anthracene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Acenaphthene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Acenaphthylene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Acetophenone | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| alpha, alpha-Dimethylphenethylamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| alpha-Terpineol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Aniline | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Anthracene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Aramite | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Atrazine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Benzaldehyde | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Benzidine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Benzo (a) anthracene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Benzo (a) pyrene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Benzo (b) fluoranthene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Benzo (ghi) perylene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Benzo (k) fluoranthene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Benzoic acid | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Benzyl alcohol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Biphenyl | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|------------------------------------|---|---|
| Bis(2-chloroethoxy) methane | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Bis(2-chloroethyl) ether | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Bis(2-chloro-1 methyl-ethyl) ether | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Bis(2-ethylhexyl) phthalate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Butyl benzyl phthalate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Caprolactam | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Carbazole | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Chlorobenzilate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Chrysene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| cis-Diallate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Diallate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Dibenzo (a,e) pyrene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Dibenzo (a,h) anthracene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Dibenzofuran | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Diethyl phthalate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Dimethoate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Dimethyl phthalate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Di-n-butyl phthalate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Di-n-octyl phthalate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Dinoseb | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Diphenylamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Disulfoton | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Ethyl methacrylate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Ethyl methanesulfonate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |



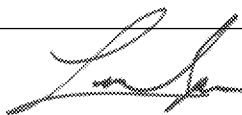
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--------------------------------|---|---|
| Famphur | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Fluoranthene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Fluorene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Hexachlorobenzene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Hexachlorobutadiene | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| Hexachlorocyclopentadiene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Hexachloroethane | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Hexachlorophene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Hydroxymethyl phthalimide | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Indeno (1,2,3-cd) pyrene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Isodrin | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Isophorone | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Isosafrole | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Kepone | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Methapyrilene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Methyl methacrylate | EPA 8270C/8270D | EPA 8270C/8270D |
| Methyl methanesulfonate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Methyl parathion | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Methylene bis(2-chloroaniline) | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Naphthalene | EPA 624.1 EPA 625.1 EPA 8310 EPA 8260B/8260C EPA 8270C/8270D ⁴ | EPA 8310 EPA 8260B/8260C EPA 8270C/8270D ⁴ |
| Nitrobenzene | EPA 625.1 EPA 8330A EPA 8270C/8270D | EPA 8330A EPA 8270C/8270D |



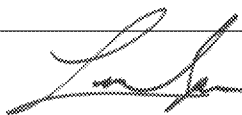
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|---------------------------------|--|---|
| Nitroquinoline-1-oxide | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| n-Decane | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| n-Nitrosodiethylamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| n-Nitrosodimethylamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| n-Nitrosodimethylethylamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| n-Nitrosodi-n-butylamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| n-Nitrosodi-n-propylamine | EPA 625.1 EPA 8270C/8270D ⁴ | EPA 8270C/8270D |
| n-Nitrosodiphenylamine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| n-Nitrosomorpholine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| n-Nitrosopiperidine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| n-Nitrosopyrrolidine | EPA 625.1 EPA 8270C/8270D ⁴ | EPA 8270C/8270D |
| n-Octadecane | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| o,o,o-Triethyl phosphorothioate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| o-Toluidine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Parathion, ethyl | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| p-Dimethylaminoazobenzene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Pentachlorobenzene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Pentachloroethane | EPA 624.1 EPA 625.1 EPA 8260B/8260C EPA 8270C/8270D | EPA 8260B/8260C EPA 8270C/8270D |
| Pentachloronitrobenzene | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Pentachlorophenol | EPA 625.1 EPA 8151A EPA 8270C/8270D | EPA 8151A EPA 8270C/8270D |
| Phenacetin | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Phenanthrene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Phenol | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |



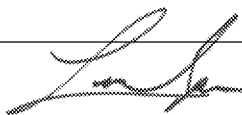
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|-------------------------------------|---|---|
| Phorate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Pronamide (Kerb) | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Pyrene | EPA 625.1 EPA 8310 EPA 8270C/8270D ⁴ | EPA 8310 EPA 8270C/8270D ⁴ |
| Pyridine | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Safrole | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Sulfotepp | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Thionazin (Zinophos) | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| trans-Diallate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| Tributyl Phosphate | EPA 625.1 EPA 8270C/8270D | EPA 8270C/8270D |
| <u>Pesticides & PCBs</u> | | |
| 2,4'-DDD | EPA 8081A/8081B | EPA 8081A/8081B |
| 2,4'-DDE | EPA 8081A/8081B | EPA 8081A/8081B |
| 2,4'-DDT | EPA 8081A/8081B | EPA 8081A/8081B |
| 4,4'-DDT | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| 4,4'-DDD | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| 4,4'-DDE | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Aldrin | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| alpha-BHC | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| beta-BHC | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Chlordane (N.O.S) | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| cis-Chlordane (alpha-Chlordane) | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| cis-Nonachlor | EPA 8081A/8081B | EPA 8081A/8081B |
| delta-BHC | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Dieldrin | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Endosulfan sulfate | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Endosulfan I | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Endosulfan II | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |



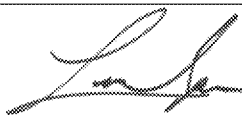
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|----------------------------------|---|---|
| Endrin | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Endrin aldehyde | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Endrin ketone | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| gamma-BHC | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Heptachlor | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Heptachlor epoxide | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Hexachlorobenzene | EPA 8081A/8081B | EPA 8081A/8081B |
| Methoxychlor | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| Mirex | EPA 8081A/8081B | EPA 8081A/8081B |
| Oxychlordane | EPA 8081A/8081B | EPA 8081A/8081B |
| Toxaphene | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| trans-Chlordane | EPA 608.3 EPA 8081A/8081B | EPA 8081A/8081B |
| trans-Nonachlor | EPA 8081A/8081B | EPA 8081A/8081B |
| PCB-1016 (Aroclor) | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| PCB-1221 | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| PCB-1232 | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| PCB-1242 | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| PCB-1248 | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| PCB-1254 | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| PCB-1260 | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| PCB-1262 | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| PCB-1268 | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| Total Aroclors | EPA 608.3 EPA 8082/8082A | EPA 8082/8082A |
| <u>FID Compounds</u> | | |
| 1,1,1-Trichloroethane | EPA 8015C/8015D | EPA 8015C/8015D |
| 2-Butanone (Methyl Ethyl Ketone) | EPA 624.1 EPA 8015C/8015D EPA 8260B/8260C | EPA 8015C/8015D EPA 8260B/8260C |
| 4-Methyl-2-Pentanone | EPA 8015C/8015D | EPA 8015C/8015D |
| Acetone | EPA 8015C/8015D | EPA 8015C/8015D |
| Benzene | EPA 8015C/8015D | EPA 8015C/8015D |



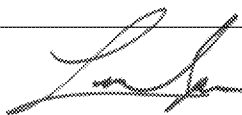
| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--|---|---|
| Chloroform | EPA 8015C/8015D | EPA 8015C/8015D |
| Diesel Range Organics (DRO) | EPA 8015C/8015D | EPA 8015C/8015D |
| Diethylene glycol | EPA 8015C/8015D | EPA 8015C/8015D |
| Ethanol | EPA 8015C/8015D | EPA 8015C/8015D |
| Ethyl acetate | EPA 624.1 EPA 8015C/8015D EPA 8260B/8260C | EPA 8015C/8015D EPA 8260B/8260C |
| Ethylbenzene | EPA 8015C/8015D | EPA 8015C/8015D |
| Ethylene glycol | EPA 8015C/8015D | EPA 8015C/8015D |
| Gas Range Organics (GRO) | EPA 8015C/8015D | EPA 8015C/8015D |
| Kerosene | EPA 8015C/8015D | EPA 8015C/8015D |
| Isobutyl alcohol | EPA 624.1 EPA 8015C/8015D EPA 8260B/8260C | EPA 8015C/8015D EPA 8260B/8260C |
| Isopropyl alcohol (2-Propanol) | EPA 8015C/8015D | EPA 8015C/8015D |
| m, p-Xylenes | EPA 8015C/8015D | EPA 8015C/8015D |
| Methanol | EPA 8015C/8015D | EPA 8015C/8015D |
| Methylene chloride | EPA 8015C/8015D | EPA 8015C/8015D |
| n-Butyl alcohol | EPA 624.1 EPA 8015C/8015D EPA 8260B/8260C | EPA 8015C/8015D EPA 8260B/8260C |
| o-Xylene | EPA 8015C/8015D | EPA 8015C/8015D |
| Propylene glycol | EPA 8015C/8015D | EPA 8015C/8015D |
| Toluene | EPA 8015C/8015D | EPA 8015C/8015D |
| Triethylene glycol | EPA 8015C/8015D | EPA 8015C/8015D |
| Volatile Petroleum Products | NWTPH-Gx(WDOE) | NWTPH-Gx(WDOE) |
| Semi-Volatile Petroleum Products | NWTPH-Dx(WDOE) | NWTPH-Dx(WDOE) |
| C8-C10 Aliphatic, Aromatic EPH | WDOE EPH | WDOE EPH |
| >C10-C12 Aliphatic, Aromatic EPH | WDOE EPH | WDOE EPH |
| >C12-C16 Aliphatic, Aromatic EPH | WDOE EPH | WDOE EPH |
| >C16-C21 Aliphatic, Aromatic EPH | WDOE EPH | WDOE EPH |
| >C21-C34 Aliphatic, Aromatic EPH | WDOE EPH | WDOE EPH |
| Alaska GRO | AK-101 (GRO) | AK-101 (GRO) |
| Alaska DRO | AK-102 (DRO) | AK-102 (DRO) |
| Alaska RRO | AK-103 (RRO) | AK-103 (RRO) |
| EPH Aliphatic C9-C18 | MADEP EPH | MADEP EPH |
| EPH Aliphatic C19-C36 | MADEP EPH | MADEP EPH |
| EPH Aromatic C11-C22 Unadjusted | MADEP EPH | MADEP EPH |
| <u>Nitrosamines, Nitroaromatics</u> | 8330A is by either LC/MS/MS or HPLC 8330B is by LC/MS/MS | |
| 1,3,5-Trinitrobenzene | EPA 625.1 EPA 8330A/8330B ⁵ EPA 8270C/8270D | EPA 8330A/8330B ⁵ EPA 8270C/8270D |
| 1,3-Dinitrobenzene | EPA 625.1 EPA 8330A/8330B ⁵ EPA 8270C/8270D | EPA 8330A/8330B ⁵ EPA 8270C/8270D |
| 2,4,6-Trinitrotoluene | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| 2,4-Dinitrotoluene | EPA 625.1 EPA 8330A/8330B ⁵ EPA 8270C/8270D | EPA 8330A/8330B ⁵ EPA 8270C/8270D |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--|--|---|
| 2,6-Dinitrotoluene | EPA 625.1 EPA 8330A/8330B ⁵ EPA 8270C/8270D | EPA 8330A/8330B ⁵ EPA 8270C/8270D |
| 2-Amino-4,6-Dinitrotoluene | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| 2-Nitrotoluene | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| <u>Nitrosamines, Nitroaromatics</u> | 8330B is by LC/MS/MS. 8330A is by either LC/MS/MS or HPLC | |
| 3,5-Dinitroaniline | EPA 8330B ⁵ | EPA 8330B ⁵ |
| 3-Nitrotoluene | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| 4-Amino-2,6-Dinitrotoluene | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| 4-Nitrotoluene | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| Nitrobenzene | EPA 625.1 EPA 8330A/8330B ⁵ EPA 8270C/8270D | EPA 8330A/8330B ⁵ EPA 8270C/8270D |
| Nitroglycerin | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7- tetrazocine (HMX) | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| Pentaerythritoltetranitrate (PETN) | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| Tetryl (methyl-2,4,6- trinitrophenylnitramine) | EPA 8330A/8330B ⁵ | EPA 8330A/8330B ⁵ |
| <u>Dissolved Gases by FID</u> | | |
| Ethane | RSK 175 | ----- |
| Ethene | RSK 175 | ----- |
| Methane | RSK 175 | ----- |
| <u>Herbicides</u> | | |
| 2,4-D | EPA 8151A | EPA 8151A |
| 2,4-DB | EPA 8151A | EPA 8151A |
| Dalapon | EPA 8151A | EPA 8151A |
| Dicamba | EPA 8151A | EPA 8151A |
| Dichloroprop | EPA 8151A | EPA 8151A |
| Dinoseb | EPA 625.1 EPA 8151A EPA 8270C/8270D | EPA 8151A EPA 8270C/8270D |
| MCPA | EPA 8151A | EPA 8151A |
| MCPP | EPA 8151A | EPA 8151A |
| 2,4,5-T | EPA 8151A | EPA 8151A |
| 2,4,5-TP (Silvex) | EPA 8151A | EPA 8151A |
| Pentachlorophenol | EPA 8151A | EPA 8151A |
| <u>Radiochemistry</u> | | |
| Barium 133 | DOE 4.5.2.3 | DOE 4.5.2.3 |
| Cesium 134 | EPA 901.1 DOE 4.5.2.3 | DOE 4.5.2.3 |
| Cesium 137 | EPA 901.1 DOE 4.5.2.3 | DOE 4.5.2.3 |



| <u>Parameter/Analyte</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste (Liquids and Solids)</u> |
|--|---|---|
| Cobalt-60 | EPA 901.1 DOE 4.5.2.3 | DOE 4.5.2.3 |
| Gamma Emitters | EPA 901.1 DOE 4.5.2.3 | DOE 4.5.2.3 |
| Gross Alpha | EPA 900.0 EPA 9310 | EPA 9310 |
| Gross Beta | EPA 900.0 EPA 9310 | EPA 9310 |
| Radioactive Iodine | EPA 901.1 EPA 902.0 DOE 4.5.2.3 | DOE 4.5.2.3 |
| Radium-226 | EPA 903.0/903.1 DOE Ra-04 | DOE Ra-04 |
| Radium-228 | EPA 904.0 EPA 9320 DOE 4.5.2.3 | EPA 9320 DOE 4.5.2.3 |
| Total Alpha Radium | EPA 903.0 EPA 9315 | EPA 9315 |
| Radon-222 | SM 7500 Rn-B | ----- |
| Strontium-89 | EPA 905.0 DOE Sr-01 | DOE Sr-01 |
| Strontium-90 | EPA 905.0 DOE Sr-02 | DOE Sr-02 |
| Thorium | EMSL-LV | EMSL-LV |
| Tritium | EPA 906.0 | EPA 906.0 Modified |
| Uranium | EPA 200.8 EPA 6020/6020A ASTM D5174-02/97 DOE U-02 | EPA 6020/6020A DOE U-02 |
| Zinc-65 | EPA 901.1 DOE 4.5.2.3 | DOE 4.5.2.3 |
| <u>Preparatory and Clean-up Methods</u> | | |
| Toxicity Characteristic Leaching Procedure (Inorganics, Extractable Organics, Volatile Organics) | EPA 1311 | EPA 1311 |
| Synthetic Precipitation Leaching Procedure | EPA 1312 | EPA 1312 |
| Waste Extraction Test (W.E.T.) | CCR Ch. 11, Article 5, Appendix II | CCR Ch. 11, Article 5, Appendix II |
| Anion Preparation | EPA 9056A ³ | EPA 9056A ³ |
| Cyanide Distillation | EPA 9010B/9010C SM 4500CN ⁻ C | EPA 9010B/9010C ³ |
| Sulfide Distillation | EPA 9030B | EPA 9030B |
| Metals Digestion | EPA 200.2 EPA 3005A EPA 3010A | EPA 3050B |
| Alkaline Digestion for Hex Chromium | ----- | EPA 3060A |
| Bomb Preparation for Solid Waste | ----- | EPA 5050 |
| Mercury Preparation | EPA 245.1/245.2 EPA 7470/7470A | EPA 7471A/7471B |



| Parameter/Analyte | Nonpotable Water | Solid Hazardous Waste (Liquids and Solids) |
|--|-------------------------|---|
| Separatory Funnel Liquid-Liquid Extraction | EPA 3510C | ----- |
| Solid Phase Extraction | EPA 3535A | EPA 3535A (Liquid) |
| Automated Soxhlet Extraction | ----- | EPA 3541 |
| Ultrasonic Extraction | ----- | EPA 3550C |
| Waste Dilution | ----- | EPA 3580A |
| Waste Dilution for Volatile Organics | ----- | EPA 3585 |
| Purge and Trap for Volatile Organics | EPA 5030A/5030B/5030C | EPA 5035/5035A/5035H/5035L |
| Alumina Clean-up | ----- | EPA 3610B EPA 3611B |
| Florisil Clean-up | EPA 3620B/3260C | EPA 3620B/3620C |
| Silica Gel Clean-up | ----- | EPA 3630C |
| Gel Permeation Clean-up | ----- | EPA 3640A |
| Sulfur Clean-up | EPA 3660B | EPA 3660B |
| Sulfuric Acid/Permanganate Clean-up | EPA 3665A | EPA 3665A |

1 – Calculated from silica determination

2 – Applicable only to liquid ‘Solid Hazardous Waste’, where liquids may include aqueous, non-aqueous, and oily wastes.

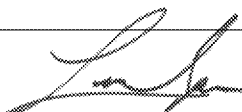
Solids may include soils, sediments, sludges, tissues, filters and any matrix deemed non-liquid.

3 – The referenced method is modified to include a simple prep for non-aqueous and/or solid matrix samples.

4 – The analytes may be determined by Selective Ion Monitoring (SIM) using either 8270C or 8270D.

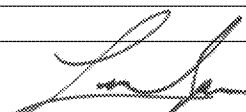
5 – 8330B analysis is performed on LC/MS/MS. 8330A may be performed on either LC/MS/MS or HPLC.

| Metals on Filters | Air Filters |
|--------------------------|-------------------------------------|
| Aluminum | EPA 6010B/6010C/6010D NIOSH 7303 |
| Antimony | EPA 6010B/6010C/6010D NIOSH 7303 |
| Arsenic | EPA 6010B/6010C/6010D NIOSH 7303 |
| Barium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Beryllium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Cadmium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Calcium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Chromium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Cobalt | EPA 6010B/6010C/6010D NIOSH 7303 |
| Copper | EPA 6010B/6010C/6010D NIOSH 7303 |
| Iron | EPA 6010B/6010C/6010D NIOSH 7303 |
| Lead | EPA 6010B/6010C/6010D NIOSH 7303 |
| Magnesium | EPA 6010B/6010C/6010D NIOSH 7303 |

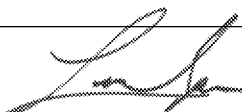


| Metals on Filters | Air Filters |
|--------------------------|-------------------------------------|
| Manganese | EPA 6010B/6010C/6010D NIOSH 7303 |
| Molybdenum | EPA 6010B/6010C/6010D NIOSH 7303 |
| Nickel | EPA 6010B/6010C/6010D NIOSH 7303 |
| Phosphorous | EPA 6010B/6010C/6010D NIOSH 7303 |
| Potassium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Selenium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Sodium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Strontium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Sulfur | EPA 6010B/6010C/6010D NIOSH 7303 |
| Tin | EPA 6010B/6010C/6010D NIOSH 7303 |
| Titanium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Uranium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Vanadium | EPA 6010B/6010C/6010D NIOSH 7303 |
| Zinc | EPA 6010B/6010C/6010D NIOSH 7303 |

| Drinking Water Organics | Drinking Water |
|------------------------------------|------------------------|
| 1,2-Dibromo-3-chloropropane (DBCP) | EPA 504.1 EPA 524.2 |
| 1,2 Dibromoethane (EDB) | EPA 504.1 EPA 524.2 |
| 1,2,3-Trichloropropane | EPA 504.1 |
| 1,4-Dioxane | EPA 522 |
| 1,1,1,2-Tetrachloroethane | EPA 524.2 |
| 1,1,1-Trichloroethane | EPA 524.2 |
| 1,1,2,2-Tetrachloroethane | EPA 524.2 |
| 1,1,2-Trichloroethane | EPA 524.2 |
| 1,1-Dichloroethane | EPA 524.2 |
| 1,1-Dichloroethene | EPA 524.2 |
| 1,1-Dichloropropene | EPA 524.2 |
| 1,2,3-Trichlorobenzene | EPA 524.2 |
| 1,2,3-Trichloropropane | EPA 524.2 |
| 1,2,4-Trichlorobenzene | EPA 524.2 |
| 1,2,4-Trimethylbenzene | EPA 524.2 |
| 1,2-Dichlorobenzene | EPA 524.2 |
| 1,2-Dichloroethane | EPA 524.2 |
| 1,2-Dichloropropane | EPA 524.2 |
| 1,3,5-Trimethylbenzene | EPA 524.2 |



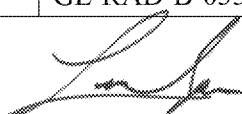
| Drinking Water Organics | Drinking Water |
|----------------------------------|----------------|
| 1,3-Dichlorobenzene | EPA 524.2 |
| 1,3-Dichloropropane | EPA 524.2 |
| 1,4-Dichlorobenzene | EPA 524.2 |
| 2,2-Dichloropropane | EPA 524.2 |
| 2-Butanone (Methyl Ethyl Ketone) | EPA 524.2 |
| 2-Chlorotoluene | EPA 524.2 |
| 2-Hexanone | EPA 524.2 |
| 4-Chlorotoluene | EPA 524.2 |
| 4-Isopropyltoluene | EPA 524.2 |
| 4-Methyl-2-pentanone | EPA 524.2 |
| Acetone | EPA 524.2 |
| Benzene | EPA 524.2 |
| Bromobenzene | EPA 524.2 |
| Bromochloromethane | EPA 524.2 |
| Bromodichloromethane | EPA 524.2 |
| Bromoform | EPA 524.2 |
| Bromomethane | EPA 524.2 |
| Carbon disulfide | EPA 524.2 |
| Carbon tetrachloride | EPA 524.2 |
| Chlorobenzene | EPA 524.2 |
| Chloroethane | EPA 524.2 |
| Chloroform | EPA 524.2 |
| Chloromethane | EPA 524.2 |
| cis-1,2-Dichloroethene | EPA 524.2 |
| cis-1,3-Dichloropropene | EPA 524.2 |
| Dibromochloromethane | EPA 524.2 |
| Dibromomethane | EPA 524.2 |
| Dichlorodifluoromethane | EPA 524.2 |
| Ethyl Benzene | EPA 524.2 |
| Hexachlorobutadiene | EPA 524.2 |
| Iodomethane | EPA 524.2 |
| Isopropylbenzene | EPA 524.2 |
| Methyl tert-butyl ether (MTBE) | EPA 524.2 |
| Methylene chloride | EPA 524.2 |
| m+p-Xylene | EPA 524.2 |
| Naphthalene | EPA 524.2 |
| n-Butylbenzene | EPA 524.2 |
| n-Propylbenzene | EPA 524.2 |
| o-Xylene | EPA 524.2 |
| Sec-Butylbenzene | EPA 524.2 |
| Styrene | EPA 524.2 |
| tert-Butylbenzene | EPA 524.2 |
| Tetrachloroethene | EPA 524.2 |
| Toluene | EPA 524.2 |
| trans-1,2-Dichloroethene | EPA 524.2 |
| trans-1,3-Dichloropropene | EPA 524.2 |
| Trichloroethene | EPA 524.2 |
| Trihalomethanes | EPA 524.2 |
| Vinyl chloride | EPA 524.2 |
| Xylenes, total | EPA 524.2 |
| Bromoacetic acid | EPA 552.2 |



| Drinking Water Organics | Drinking Water |
|--------------------------------|-----------------------|
| Bromochloroacetic acid | EPA 552.2 |
| Chloroacetic acid | EPA 552.2 |
| Dibromoacetic acid | EPA 552.2 |
| Dichloroacetic acid | EPA 552.2 |
| Trichloroacetic acid | EPA 552.2 |

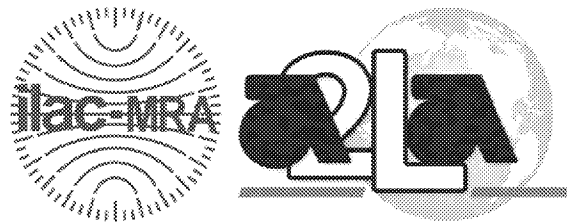
Additionally, in recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with the 2009 TNI Environmental Testing Laboratory Standard Requirements), accreditation is granted to this laboratory to perform the following bioassay analyses on bone, tissue, urine, fecal, and nasal swabs.

| <u>Bioassay Analysis(s)</u> | <u>Preparation SOP(s)</u> | <u>Analytical SOP(s)</u> |
|---|---|--|
| <u>Alpha Spectrometry:</u> Alpha: Am-241, Cm-242, Cm-243/244, Cm 245/246, Cf-252, Np-237, Po-208, Po-209, Po-210, Pu-236, Pu-238, Pu-239/240, Pu-242, Pu-244, Ra-224, Ra-226, Th-228, Th-229, Th-230, Th-232, U-232, U-233/234, U-235/236, U-238 | GL-RAD-B-001, GL-RAD-B-002, GL-RAD-B-003, GL-RAD-B-010, GL-RAD-B-012, GL-RAD-B-013, GL-RAD-B-017, GL-RAD-B-038, GL-RAD-B-040, GL-RAD-B-041 GL-RAD-B-042 | GL-RAD-B-009 |
| <u>Liquid Scintillation Spectrometry:</u> C-14, Fe-55, Gross Alpha, H-3, Ni-59, Ni-63, Pu-241, Tc-99 | GL-RAD-B-001, GL-RAD-B-008, GL-RAD-B-011, GL-RAD-B-012, GL-RAD-B-013, GL-RAD-B-016, GL-RAD-B-020, GL-RAD-B-023 | GL-RAD-I-004, GL-RAD-I-014, GL-RAD-I-017 |
| <u>Gas Flow Proportional Counting:</u> Beta: Sr-90 | GL-RAD-B-001 | GL-RAD-I-006, GL-RAD-I-015, GL-RAD-I-016 |
| <u>Bioassay Analysis(s)</u> | <u>Preparation SOP(s)</u> | <u>Analytical SOP(s)</u> |
| Gross Alpha/Gross Beta | GL-RAD-B-022 | GL-RAD-I-006 |
| <u>Kinetic Phosphorescence Analyzer:</u> Total Uranium | GL-RAD-B-019 | GL-RAD-B-018 |
| <u>Radon Emanation:</u> Ra-226 | GL-RAD-B-002 | GL-RAD-I-007 |
| <u>Refractometer:</u> Specific Gravity | GL-RAD-B-027 | GL-RAD-B-027 |
| <u>ICP-MS:</u> Uranium Isotopes | GL-RAD-B-035 | GL-RAD-B-034 |



| | | |
|---|-------------------------------|--------------|
| Gamma Spectrometry: Gamma: Ni-59, 46 to 1836 keV | GL-RAD-B-020, GL-RAD-A-013 | GL-RAD-I-001 |
|---|-------------------------------|--------------|

A handwritten signature in black ink, appearing to be 'L. L. L.', is located in the bottom center of the page.



Accredited Laboratory

A2LA has accredited

GEL LABORATORIES, LLC

Charleston, SC

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in Version 5.1 of the DoD Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).



Presented this 30th day of August 2017.

A handwritten signature in black ink, appearing to be 'L. L. L.', written over a horizontal line.

President and CEO
For the Accreditation Council
Certificate Number 2567.01
Valid to June 30, 2019
Revised June 26, 2018

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.



STATE WATER RESOURCES CONTROL BOARD
REGIONAL WATER QUALITY CONTROL BOARDS

CALIFORNIA STATE



ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

CERTIFICATE OF ENVIRONMENTAL ACCREDITATION

Is hereby granted to

GEL Laboratories, LLC

2040 Savage Road
Charleston, SC 29407

Scope of the certificate is limited to the
"Fields of Testing"
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site inspection,
proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of
Section 100825, et seq. of the Health and Safety Code.

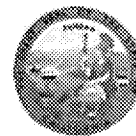
Certificate No.: 2940

Expiration Date: 10/31/2018

Effective Date: 12/1/2017

Sacramento, California
subject to forfeiture or revocation

Christine Sotelo, Chief
Environmental Laboratory Accreditation Program



GEL Laboratories, LLC

2040 Savage Road
Charleston, SC 29407
Phone: (843) 556-8171

Certificate No. 2940
Expiration Date 10/31/2018

Field of Testing: 106 - Radiochemistry of Drinking Water

| | | | |
|---------|-----|--------------------------------|---------------|
| 106.010 | 001 | Gross Alpha and Beta Radiation | EPA 900.0 |
| 106.010 | 002 | Gross Beta | EPA 900.0 |
| 106.030 | 001 | Radioactive Cesium | EPA 901.1 |
| 106.030 | 002 | Radioactive Iodine | EPA 901.1 |
| 106.030 | 003 | Gamma Emitters | EPA 901.1 |
| 106.040 | 001 | Radioactive Iodine | EPA 902.0 |
| 106.050 | 001 | Total Alpha Radium | EPA 903.0 |
| 106.050 | 002 | Radium-226 (estimate) | EPA 903.0 |
| 106.051 | 001 | Radium-226 | EPA 903.1 |
| 106.060 | 001 | Radium-228 | EPA 904.0 |
| 106.070 | 001 | Strontium-89, 90 | EPA 905.0 |
| 106.070 | 002 | Strontium-89 | EPA 905.0 |
| 106.070 | 003 | Strontium-90 | EPA 905.0 |
| 106.080 | 001 | Tritium | EPA 906.0 |
| 106.092 | 001 | Uranium | EPA 200.8 |
| 106.120 | 001 | Gross Alpha by Coprecipitation | EPA 00-02 |
| 106.230 | 001 | Isotopic Uranium | DOE U-02 |
| 106.250 | 002 | Radioactive Iodine | DOE 4.5.2.3 |
| 106.250 | 003 | Gamma Emitters | DOE 4.5.2.3 |
| 106.480 | 001 | Uranium (mass) | ASTM D5174-97 |

Field of Testing: 108 - Inorganic Chemistry of Wastewater

| | | | |
|---------|-----|------------------------|-----------|
| 108.020 | 001 | Conductivity | EPA 120.1 |
| 108.090 | 001 | Residue, Volatile | EPA 160.4 |
| 108.110 | 001 | Turbidity | EPA 180.1 |
| 108.112 | 001 | Boron | EPA 200.7 |
| 108.112 | 002 | Calcium | EPA 200.7 |
| 108.112 | 003 | Hardness (calculation) | EPA 200.7 |
| 108.112 | 004 | Magnesium | EPA 200.7 |
| 108.112 | 005 | Potassium | EPA 200.7 |
| 108.112 | 006 | Silica, Dissolved | EPA 200.7 |
| 108.112 | 007 | Sodium | EPA 200.7 |
| 108.113 | 001 | Boron | EPA 200.8 |
| 108.113 | 002 | Calcium | EPA 200.8 |
| 108.113 | 003 | Magnesium | EPA 200.8 |
| 108.120 | 001 | Bromide | EPA 300.0 |
| 108.120 | 002 | Chloride | EPA 300.0 |
| 108.120 | 003 | Fluoride | EPA 300.0 |
| 108.120 | 008 | Sulfate | EPA 300.0 |

| | | | |
|---------|-----|---------------------------------|-----------------------|
| 108.120 | 012 | Nitrate (as N) | EPA 300.0 |
| 108.120 | 013 | Nitrate-Nitrite (as N) | EPA 300.0 |
| 108.120 | 014 | Nitrite (as N) | EPA 300.0 |
| 108.120 | 015 | Phosphate, Ortho (as P) | EPA 300.0 |
| 108.183 | 001 | Cyanide, Total | EPA 335.4 |
| 108.209 | 001 | Ammonia (as N) | EPA 350.1 |
| 108.211 | 002 | Kjeldahl Nitrogen, Total (as N) | EPA 351.2 |
| 108.232 | 004 | Nitrite (as N) | EPA 353.2 |
| 108.266 | 001 | Phosphorus, Total | EPA 365.4 |
| 108.323 | 001 | Chemical Oxygen Demand | EPA 410.4 |
| 108.362 | 001 | Phenols, Total | EPA 420.4 |
| 108.381 | 001 | Oil and Grease | EPA 1664A |
| 108.385 | 001 | Color | SM2120B-2001 |
| 108.390 | 001 | Turbidity | SM2130B-2001 |
| 108.400 | 001 | Acidity | SM2310B-1997 |
| 108.410 | 001 | Alkalinity | SM2320B-1997 |
| 108.420 | 001 | Hardness (calculation) | SM2340B-1997 |
| 108.421 | 001 | Hardness | SM2340C-1997 |
| 108.430 | 001 | Conductivity | SM2510B-1997 |
| 108.439 | 001 | Residue, Volatile | SM2540E-1997 |
| 108.440 | 001 | Residue, Total | SM2540B-1997 |
| 108.441 | 001 | Residue, Filterable TDS | SM2540C-1997 |
| 108.442 | 001 | Residue, Non-filterable TSS | SM2540D-1997 |
| 108.465 | 001 | Chlorine, Total Residual | SM4500-Cl G-2000 |
| 108.470 | 001 | Cyanide, Total | SM4500-CN B or C-1999 |
| 108.472 | 001 | Cyanide, Total | SM4500-CN E-1999 |
| 108.473 | 001 | Cyanide, amenable | SM4500-CN G-1999 |
| 108.490 | 001 | Hydrogen Ion (pH) | SM4500-H+ B-2000 |
| 108.501 | 002 | Kjeldahl Nitrogen, Total (as N) | SM4500-NH3 C-1997 |
| 108.508 | 002 | Ammonia (as N) | SM4500-NH3 G-1997 |
| 108.513 | 001 | Kjeldahl Nitrogen, Total (as N) | SM4500-Norg D-1997 |
| 108.529 | 002 | Nitrite (as N) | SM4500-NO3- F-2000 |
| 108.545 | 001 | Phosphorus, Total | SM4500-P H-1999 |
| 108.560 | 001 | Sulfite | SM4500-SO3 B-2000 |
| 108.584 | 001 | Sulfide (as S) | SM4500-S= D-2000 |
| 108.592 | 001 | Biochemical Oxygen Demand | SM5210B-2001 |
| 108.592 | 002 | Carbonaceous BOD | SM5210B-2001 |
| 108.595 | 001 | Chemical Oxygen Demand | SM5220D-1997 |
| 108.596 | 001 | Organic Carbon-Total (TOC) | SM5310B-2000 |

Field of Testing: 109 - Toxic Chemical Elements of Wastewater

| | | | |
|---------|-----|-----------|-----------|
| 109.010 | 001 | Aluminum | EPA 200.7 |
| 109.010 | 002 | Antimony | EPA 200.7 |
| 109.010 | 003 | Arsenic | EPA 200.7 |
| 109.010 | 004 | Barium | EPA 200.7 |
| 109.010 | 005 | Beryllium | EPA 200.7 |
| 109.010 | 006 | Boron | EPA 200.7 |
| 109.010 | 007 | Cadmium | EPA 200.7 |

As of 12/14/2017, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

| | | | |
|---------|-----|---------------|------------------|
| 109.010 | 009 | Chromium | EPA 200.7 |
| 109.010 | 010 | Cobalt | EPA 200.7 |
| 109.010 | 011 | Copper | EPA 200.7 |
| 109.010 | 012 | Iron | EPA 200.7 |
| 109.010 | 013 | Lead | EPA 200.7 |
| 109.010 | 015 | Manganese | EPA 200.7 |
| 109.010 | 016 | Molybdenum | EPA 200.7 |
| 109.010 | 017 | Nickel | EPA 200.7 |
| 109.010 | 019 | Selenium | EPA 200.7 |
| 109.010 | 021 | Silver | EPA 200.7 |
| 109.010 | 023 | Thallium | EPA 200.7 |
| 109.010 | 024 | Tin | EPA 200.7 |
| 109.010 | 025 | Titanium | EPA 200.7 |
| 109.010 | 026 | Vanadium | EPA 200.7 |
| 109.010 | 027 | Zinc | EPA 200.7 |
| 109.020 | 001 | Aluminum | EPA 200.8 |
| 109.020 | 002 | Antimony | EPA 200.8 |
| 109.020 | 003 | Arsenic | EPA 200.8 |
| 109.020 | 004 | Barium | EPA 200.8 |
| 109.020 | 005 | Beryllium | EPA 200.8 |
| 109.020 | 006 | Cadmium | EPA 200.8 |
| 109.020 | 007 | Chromium | EPA 200.8 |
| 109.020 | 008 | Cobalt | EPA 200.8 |
| 109.020 | 009 | Copper | EPA 200.8 |
| 109.020 | 010 | Lead | EPA 200.8 |
| 109.020 | 011 | Manganese | EPA 200.8 |
| 109.020 | 012 | Molybdenum | EPA 200.8 |
| 109.020 | 013 | Nickel | EPA 200.8 |
| 109.020 | 014 | Selenium | EPA 200.8 |
| 109.020 | 015 | Silver | EPA 200.8 |
| 109.020 | 016 | Thallium | EPA 200.8 |
| 109.020 | 017 | Vanadium | EPA 200.8 |
| 109.020 | 018 | Zinc | EPA 200.8 |
| 109.020 | 021 | Iron | EPA 200.8 |
| 109.020 | 022 | Tin | EPA 200.8 |
| 109.190 | 001 | Mercury | EPA 245.1 |
| 109.191 | 001 | Mercury | EPA 245.2 |
| 109.361 | 001 | Mercury | EPA 1631E |
| 109.445 | 002 | Chromium (VI) | SM3500-Cr B-2009 |

Field of Testing: 110 - Volatile Organic Chemistry of Wastewater

| | | | |
|---------|-----|-----------------------------|---------|
| 110.040 | 000 | Purgeable Organic Compounds | EPA 624 |
|---------|-----|-----------------------------|---------|

Field of Testing: 111 - Semi-volatile Organic Chemistry of Wastewater

| | | | |
|---------|-----|------------------------------------|---------|
| 111.100 | 000 | Base/Neutral & Acid Organics | EPA 625 |
| 111.170 | 000 | Organochlorine Pesticides and PCBs | EPA 608 |

Field of Testing: 112 - Radiochemistry of Wastewater

| | | | |
|---------|-----|--------------------------------|-----------|
| 112.010 | 001 | Gross Alpha and Beta Radiation | EPA 900.0 |
|---------|-----|--------------------------------|-----------|

As of 12/14/2017, this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

| | | | |
|---------|-----|--------------------|-------------|
| 112.010 | 002 | Gross Beta | EPA 900.0 |
| 112.020 | 001 | Total Alpha Radium | EPA 903.0 |
| 112.021 | 001 | Radium-226 | EPA 903.1 |
| 112.140 | 001 | Cesium | EPA 901.1 |
| 112.140 | 002 | Gamma | EPA 901.1 |
| 112.160 | 001 | Radium-228 | EPA 904.0 |
| 112.170 | 001 | Strontium | EPA 905.0 |
| 112.180 | 001 | Tritium | EPA 906.0 |
| 112.490 | 001 | Cesium | DOE 4.5.2.3 |
| 112.490 | 002 | Gamma Emitters | DOE 4.5.2.3 |
| 112.500 | 001 | Strontium | DOE Sr-01 |
| 112.510 | 001 | Strontium | DOE Sr-02 |
| 112.520 | 001 | Isotopic Uranium | DOE U-02 |

Field of Testing: 114 - Inorganic Chemistry of Hazardous Waste

| | | | |
|---------|-----|---------------|-----------|
| 114.010 | 001 | Antimony | EPA 6010B |
| 114.010 | 002 | Arsenic | EPA 6010B |
| 114.010 | 003 | Barium | EPA 6010B |
| 114.010 | 004 | Beryllium | EPA 6010B |
| 114.010 | 005 | Cadmium | EPA 6010B |
| 114.010 | 006 | Chromium | EPA 6010B |
| 114.010 | 007 | Cobalt | EPA 6010B |
| 114.010 | 008 | Copper | EPA 6010B |
| 114.010 | 009 | Lead | EPA 6010B |
| 114.010 | 010 | Molybdenum | EPA 6010B |
| 114.010 | 011 | Nickel | EPA 6010B |
| 114.010 | 012 | Selenium | EPA 6010B |
| 114.010 | 013 | Silver | EPA 6010B |
| 114.010 | 014 | Thallium | EPA 6010B |
| 114.010 | 015 | Vanadium | EPA 6010B |
| 114.010 | 016 | Zinc | EPA 6010B |
| 114.020 | 001 | Antimony | EPA 6020 |
| 114.020 | 002 | Arsenic | EPA 6020 |
| 114.020 | 003 | Barium | EPA 6020 |
| 114.020 | 004 | Beryllium | EPA 6020 |
| 114.020 | 005 | Cadmium | EPA 6020 |
| 114.020 | 006 | Chromium | EPA 6020 |
| 114.020 | 007 | Cobalt | EPA 6020 |
| 114.020 | 008 | Copper | EPA 6020 |
| 114.020 | 009 | Lead | EPA 6020 |
| 114.020 | 010 | Molybdenum | EPA 6020 |
| 114.020 | 011 | Nickel | EPA 6020 |
| 114.020 | 012 | Selenium | EPA 6020 |
| 114.020 | 013 | Silver | EPA 6020 |
| 114.020 | 014 | Thallium | EPA 6020 |
| 114.020 | 015 | Vanadium | EPA 6020 |
| 114.020 | 016 | Zinc | EPA 6020 |
| 114.103 | 001 | Chromium (VI) | EPA 7196A |

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| | | | |
|---------|-----|--------------------------------|-----------|
| 114.141 | 001 | Mercury | EPA 7471A |
| 114.221 | 001 | Cyanide, Total | EPA 9012A |
| 114.230 | 001 | Sulfides, Total | EPA 9034 |
| 114.241 | 001 | Corrosivity - pH Determination | EPA 9045C |
| 114.250 | 001 | Fluoride | EPA 9056 |

Field of Testing: 115 - Extraction Test of Hazardous Waste

| | | | |
|---------|-----|---|--|
| 115.020 | 001 | Toxicity Characteristic Leaching Procedure (TCLP) | EPA 1311 |
| 115.021 | 001 | TCLP Inorganics | EPA 1311 |
| 115.022 | 001 | TCLP Extractables | EPA 1311 |
| 115.023 | 001 | TCLP Volatiles | EPA 1311 |
| 115.030 | 001 | Waste Extraction Test (WET) | CCR Chapter 11, Article 5, Appendix II |
| 115.040 | 001 | Synthetic Precipitation Leaching Procedure (SPLP) | EPA 1312 |

Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste

| | | | |
|---------|-----|----------------------------|-----------|
| 116.010 | 000 | EDB and DBCP | EPA 8011 |
| 116.020 | 030 | Nonhalogenated Volatiles | EPA 8015B |
| 116.020 | 031 | Ethanol and Methanol | EPA 8015B |
| 116.030 | 001 | Gasoline-range Organics | EPA 8015B |
| 116.080 | 000 | Volatile Organic Compounds | EPA 8260B |
| 116.080 | 120 | Oxygenates | EPA 8260B |

Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste

| | | | |
|---------|-----|---|-----------|
| 117.010 | 001 | Diesel-range Total Petroleum Hydrocarbons | EPA 8015B |
| 117.110 | 000 | Extractable Organics | EPA 8270C |
| 117.140 | 000 | Polynuclear Aromatic Hydrocarbons | EPA 8310 |
| 117.170 | 000 | Nitroaromatics and Nitramines | EPA 8330 |
| 117.171 | 000 | Nitroaromatics and Nitramines | EPA 8330A |
| 117.210 | 000 | Organochlorine Pesticides | EPA 8081A |
| 117.220 | 000 | PCBs | EPA 8082 |
| 117.250 | 000 | Chlorinated Herbicides | EPA 8151A |

Field of Testing: 118 - Radiochemistry of Hazardous Waste

| | | | |
|---------|-----|------------------|------------------------|
| 118.010 | 001 | Gross Alpha | EPA 9310 |
| 118.010 | 002 | Gross Beta | EPA 9310 |
| 118.020 | 001 | Radium, Total | EPA 9315 |
| 118.030 | 001 | Radium-228 | EPA 9320 |
| 118.070 | 001 | Thorium | EPA (March, 1979), p33 |
| 118.140 | 001 | Radium-226 | EPA Ra-04 |
| 118.200 | 001 | Gamma Emitters | DOE 4.5.2.3 |
| 118.270 | 001 | Strontium | DOE Sr-01 |
| 118.271 | 001 | Strontium | DOE Sr-02 |
| 118.290 | 001 | Isotopic Uranium | DOE U-02 |

Field of Testing: 120 - Physical Properties of Hazardous Waste

| | | | |
|---------|-----|--------------------------------|--------------------|
| 120.020 | 001 | Ignitability | EPA 1020A |
| 120.040 | 001 | Reactive Cyanide | Section 7.3 SW-846 |
| 120.080 | 001 | Corrosivity - pH Determination | EPA 9045C |



STATE WATER RESOURCES CONTROL BOARD
REGIONAL WATER QUALITY CONTROL BOARDS

CALIFORNIA STATE



ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

CERTIFICATE OF ENVIRONMENTAL ACCREDITATION

Is hereby granted to

GEL Laboratories, LLC

2040 Savage Road

Charleston, SC 29407

Scope of the certificate is limited to the
"Fields of Testing"
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site inspection,
proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **2940**

Expiration Date: **10/31/2018**

Effective Date: **12/1/2017**

Sacramento, California
subject to forfeiture or revocation

Christine Sotelo, Chief
Environmental Laboratory Accreditation Program

Attachment 5

Technical Systems Audit Checklist

Technical Systems Audit Checklist

| | | | |
|-----------------|----------------|---------------|-------|
| Project No. | _____ | Date of Audit | _____ |
| Project Manager | _____ | Auditor | _____ |
| Facility Name | _____ | | |
| Location | _____ | | |
| Weather | _____ | | |
| Field Personnel | FTL: _____ | SSHO: _____ | |
| | Chemist: _____ | | |
| | _____ | | |
| | _____ | | |
| | _____ | | |

Description of Field Activities

Summary and Recommendations

Planning and Preparation

| | | <u>Yes or No</u> | |
|----|---|--------------------------|--------------------------|
| 1. | Was the field audit announced or unannounced? Comments: _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 2. | Was a QA Project Plan prepared for this activity? Comments: _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 3. | Was a site Health and Safety Plan prepared for this activity? Comments: _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 4. | Were project instructions, work plan, and contractor SOWs distributed to the team? Comments: _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 5. | Were additional instructions given to project field participants (i.e., changes in project plan)? Comments: _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 6. | Was there a written list of sampling locations and descriptions? Comments: _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 7. | Was there a map of sampling locations available to field personnel? Comments: _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |

Planning and Preparation (continued)

Yes or No

- | | | | |
|-----|--|--------------------------|--------------------------|
| 8. | Was equipment list given to equipment coordinator with adequate lead time? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 9. | Was laboratory given a list of sample containers with adequate lead time? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 10. | Were analyses scheduled with the laboratory in advance? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 11. | Was the project team provided with a contact list (names & phone #s)? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 12. | Are inexperienced or poorly trained staff receiving adequate training and supervision? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 13. | Was State "One Call" agency contacted prior to drilling, trenching, or excavation to identify buried utilities? If yes, record Ticket No. _____, date of request _____, and renewal date _____ Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 14. | Was an underground utility location contractor retained to identify buried utilities? Describe means used to track and verify completion of location activities at each sampling station: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |

Planning and Preparation (continued)

- | | | <u>Yes or No</u> | |
|----|---|--------------------------|--------------------------|
| 15 | Are dig permits required? If yes, record permit number: _____ date of issue _____, and renewal date _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 16 | Are hot work permits required? If yes, record permit number _____, date and time of issue _____, and expiration date and time _____ | <input type="checkbox"/> | <input type="checkbox"/> |

Monitoring Well Installation

- | | | <u>Yes or No</u> | |
|---|---|--|--|
| 1 | Was a daily tail gate safety briefing conducted? If yes, list the items discussed: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 2 | Were the wells located properly with respect to potential contaminant plumes? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 3 | If field conditions mandated selection of a new location, was the new location properly selected? If yes, were the reasons for the relocation properly documented? | <input type="checkbox"/> | <input type="checkbox"/> |
| 4 | Were the well locations surveyed? Were the exact elevations determined as part of the survey? Were elevations referenced to a bench mark? Were horizontal coordinates established? | <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> | <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> |
| 5 | Describe the drilling techniques used. _____ _____ _____ | | |
| 6 | Was all in-ground drilling equipment properly decontaminated before initial use and between drilling locations? | <input type="checkbox"/> | <input type="checkbox"/> |

Monitoring Well Installation (continued)

Yes or No

- 7 Describe decontamination procedures (steam cleaner, pressure washer, type of soap used if any, solvent, etc.)

- 8 How was this equipment stored or otherwise protected after decontamination to prevent recontamination prior to use?

- 9 What types of casing/screen material were used (black iron, stainless steel, PVC, etc.)?

- 10 Were well casings/screens properly decontaminated before use?
Describe decontamination procedure.

☐
☐

- 11 How was this equipment stored or otherwise protected after decontamination to prevent recontamination prior to use?

- 12 Were the wells completed to the proper depth?
Were the wells screened at the proper interval?
Comments: _____

☐
☐
☐
☐

- 13 Were the newly installed wells properly secured (sealed) during the overnight grout curing required before installation of protective outer casing?

☐
☐

Monitoring Well Installation (continued)

Yes or No

- | | | | |
|----|--|--------------------------|--------------------------|
| 14 | Was a locking cap or some other locking mechanism included as part of the protective outer casing? | <input type="checkbox"/> | <input type="checkbox"/> |
| 15 | Describe disposal/storage method used for drilling mud and cuttings. _____ _____ _____ | | |
| 16 | Were samples of drilling mud, sand pack, gravel, grout, etc., collected for analysis? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 17 | Were the wells developed? If yes, describe method used _____ _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 18 | Did the drilling personnel follow required safety protocols? Comments: _____ _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |

Sampling

General Procedures

Yes or No

- | | | | |
|----|--|--------------------------|--------------------------|
| 1. | Were sampling locations properly selected? Comments: _____ _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
|----|--|--------------------------|--------------------------|

General Procedures (continued)

Yes or No

- | | | | |
|----|---|--------------------------|--------------------------|
| 2. | <p>Were samples collected starting with the least likely contaminated and proceeding to the most likely contaminated?</p> <p>Comments: _____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 3. | <p>Were new disposable gloves worn during sample collection?</p> <p>Comments: _____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 4. | <p>Was sampling equipment wrapped in aluminum foil or otherwise protected from possible contamination prior to sample collection?</p> <p>Comments: _____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 5. | <p>If equipment was cleaned in the field, were proper procedures used? (This includes storage method for rinse water and solvents.)</p> <p>Comments: _____</p> <p>_____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 6. | <p>What field instruments were used during this investigation?</p> <p>_____</p> <p>_____</p> <p>_____</p> | | |
| 7. | <p>Were field instruments properly calibrated?</p> <p>Comments: _____</p> <p>_____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 8. | <p>Were calibration procedures documented in the field notes?</p> <p>Comments: _____</p> <p>_____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |

General Procedures (continued)**Yes or No**

9. Were samples chemically field preserved?

☐☐

Comments: _____

10. Were samples iced?

☐☐

Comments: _____

11. Were samples of drilling mud, sand pack, gravel, grout, etc., collected for analysis?

☐☐

If yes, please list parameters and procedures.

Groundwater Sampling**Yes or No**

1. Was depth of well determined?

☐☐

Comments: _____

2. Was depth to water determined?

☐☐

Comments: _____

3. Was measuring tape properly decontaminated between wells?

☐☐

Comments: _____

4. Were the above depths to water converted to water level elevations common to all wells? Describe how the depths were determined.

☐☐

Groundwater Sampling (continued)

Yes or No

5. How was the volume of water originally present in each well determined?

Comments: _____

6. Was the volume determined correctly?

☐ ☐

7. How was completeness of purging determined?

Volume ☐
 Measure ☐
 Time/Flow Rate ☐
 Cond./pH/T ☐

8. Was a sufficient volume purged?

☐ ☐

9. Describe the disposal of purge water.

10. Was a dedicated (in-place) pump utilized?

☐ ☐

If no, describe the method of purging (bailer - include type and construction material, pump - include type).

11. How were the samples collected?

| | | | |
|--------|--------------------------|---------------------------------------|--------------------------|
| Bailer | <input type="checkbox"/> | Passive diffusion bag | <input type="checkbox"/> |
| Pump | <input type="checkbox"/> | Peristaltic/Bladder/Centrifugal/Other | (check one) |
| Other | <input type="checkbox"/> | | |

Groundwater Sampling (continued)

Yes or No

- 12 Construction material of bailer or tubing: Design of bailer:
- | | | | |
|--------|--------------------------|------------|--------------------------|
| S.S. | <input type="checkbox"/> | | |
| Teflon | <input type="checkbox"/> | Open top | <input type="checkbox"/> |
| PVC | <input type="checkbox"/> | Closed top | <input type="checkbox"/> |
| Other | <input type="checkbox"/> | | |

Comments: _____

- 13 If a pump was used, describe how it was cleaned before and/or between wells?
- _____
- _____
- _____

- 14 Were the samples properly transferred from bailer to sample bottles (i.e., was the purgeable sample agitated, etc.)? ☐ ☐

Comments: _____

- 15 Was the rope or line allowed to touch the ground? ☐ ☐

- 16 Was the wetted rope or line discarded after use at each well? ☐ ☐
- _____

Surface Water Sampling

Yes or No

1. What procedures were used to collect surface water?
- _____
- _____

2. Did the samplers wade in the stream during sample collection? ☐ ☐
- If yes, did sampler face upstream while collecting sample? ☐ ☐

Surface Water Sampling (continued)

Yes or No

- 3 Did the sampler insure that disturbed sediments were not collected along with water sample? ☐ ☐
- 4 Note any deficiencies observed during the collection of the surface water samples.

Soil/Sediment Sampling

1. What procedures (including equipment) were used to collect the samples?
- ---
2. Were the samples well mixed prior to placing the sample in the sample container? ☐ ☐
3. Were samples for purgeable organics analysis collected prior to mixing? ☐ ☐
4. Were samples composited?
If so, how were composites collected and mixed? ☐ ☐
- ---
5. Note any deficiencies observed during the collection of the samples.

Other Sampling

Yes or No

1. What other types of samples were collected during this investigation?

2. What procedures were used for the collection of these samples?

3. Note any deficiencies observed during the collection of these samples.

Quality Assurance/Quality Control

Yes or No

(While not all of these QA/QC procedures will be necessary during each sampling activity, the following techniques may be employed. If so, please note.)

1. Did sampling personnel utilize trip blanks? ☐ ☐
2. Did sampling personnel utilize preservative blanks? ☐ ☐
If yes, to either of the above questions, list the types and handling of the blanks.

3. Were any equipment blanks collected? ☐ ☐
If yes, list.

4. Was the water for field blank preparation appropriate for the parameter coverage? ☐ ☐
Comments: _____

5. Were any duplicate samples collected? ☐ ☐
If yes, list the types (parameter coverage, etc.) and describe their handling.

6. Were any spiked samples collected? ☐ ☐
If yes, list the types (parameter coverage, etc.) and describe their handling.

7. Were the QA/QC samples collected in accordance with the QA Project Plan? ☐ ☐
8. Were staff aware of sample holding times? ☐ ☐

Field Documentation and Chain-of-Custody

| | | <u>Yes or No</u> | |
|----|---|--------------------------|--------------------------|
| 1. | <p>Were Sample I.D. Tags filled out completely (i.e., station no., location, date, time, analyses, signatures of samplers, type of preservative)?</p> <p>Comments: _____</p> <p>_____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 2. | <p>Were Chain-of-Custody Records completed for all samples?</p> <p>Comments: _____</p> <p>_____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 3. | <p>Did information on Sample I.D. Tags and Chain-of-Custody Records match?</p> <p>Comments: _____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 4. | <p>Were samples shipped to the laboratory?</p> <p>If yes, did the Chain-of-Custody Record indicate the method of sample shipment?</p> <p>_____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 5. | <p>Was a Chain-of-Custody Record included with the samples in the shipping container?</p> <p>_____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |
| 6. | <p>Were samples properly secured to maintain custody after collection?</p> <p>Comments: _____</p> <p>_____</p> <p>_____</p> | <input type="checkbox"/> | <input type="checkbox"/> |

Field Documentation and Chain-of-Custody (continued)

Yes or No

- | | | | |
|----|--|--------------------------|--------------------------|
| 7 | Were sample tags, Chain-of-Custody forms, and field notebook signed by sampling personnel? Comments: _____ _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 8 | Does the field notebook contain adequate information about each sample including the sample I.D. number, date, location, and information necessary to reconstruct the sample? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 9 | Were entries to the field notebook made in ink? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 10 | Were corrections properly executed with one line through the error in the field notebook? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |
| 11 | Was sampling documented with photographs? If yes, was a photolog maintained? | <input type="checkbox"/> | <input type="checkbox"/> |
| 12 | Were amendments to the project plan documented (on the project plan itself, in a project logbook, elsewhere)? Comments: _____ _____ _____ | <input type="checkbox"/> | <input type="checkbox"/> |

Debriefing Following Field Audit

Yes or No

1. Was a debriefing held with project participants after the audit was completed? ☐ ☐

Comments: _____

2. Were any recommendations made to project participants during the debriefing? ☐ ☐

If yes, briefly describe recommendations made.

This image shows a single sheet of white paper with horizontal blue ruling lines. The lines are evenly spaced and run across the width of the page. There are no margins, text, or other markings on the paper.